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# HIGH TEMPERATURE POLYIMIDE FOAMS FOR SHUTTLE UPPER SURFACE THERMAL INSULATION

by George L. Ball III, James W. Leffingwell,  
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MONSANTO RESEARCH CORPORATION

Prepared Under Contract NAS1-12990

For

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION  
Washington, DC 20546

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## ABSTRACT

Polyimide foams developed by Monsanto Company and refined under NASA Contract NAS9-12246 were examined for use as upper surface space shuttle thermal insulation. It was found that postcured polyimide foams having a density of  $64 \text{ kg/m}^3$  ( $4 \text{ lb/ft}^3$ ) had acceptable physical properties up to and exceeding  $700^\circ\text{K}$  ( $800^\circ\text{F}$ ). Physical tests included cyclic heating and cooling in vacuum, weight and dimensional stability, mechanical strength and impact resistance, acoustic loading and thermal conductivity. Molding and newly developed postcuring procedures were defined.

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## SUMMARY

Polyimide foams, based on commercially available foamable polyimide powders\* and a process developed by Monsanto Research Corporation under contract to the NASA Manned Spacecraft Center (NAS9-12246), were examined for their utility as thermal insulating materials for the upper surface of the space shuttle.

Foams having densities from 32 to 128 kg/m<sup>3</sup> (2-8 lb/ft<sup>3</sup>) were examined for utility up to 725°K (850°F). The basic polyimide foam is molded at 600°K (625°F) and was found to be useful up to that temperature without any further modification. Postcuring of the foams at or above higher expected use temperatures was found to be necessary and useful. An incremental postcuring treatment up to 700°K (800°F) was established.

Utility of the polyimide foam was shown to extend to as high as 725°K (850°F) for periods of less than 10 hours, following the postcuring treatment. The anticipated usable life (in hours) of the polyimide foam in the shuttle application is illustrated in Figure 1 as a function of temperature. These lives were extrapolated from actual data on shrinkage of polyimide foams soaked throughout at the indicated temperatures. A linear loss of 5% (decrease in width or length of 5%) was taken to be the criteria of useful life, since excessive shrinkage could lead to panel separation. The difference shown in Figure 1 is a result of the fact that only the surface of the foam would be at the indicated temperature in the shuttle application. Thus, the interior of the foam would be subjected to significantly lower temperatures. It was shown that the temperature differential across a 2.3 cm (0.91 in.) thickness of the foam would be at least 225K° (400F°) at a surface temperature of 600°K (625°F).

The optimum density for the polyimide foam was shown to be 64 kg/m<sup>3</sup> (4 lb/ft<sup>3</sup>). Foams of lower densities exhibited inadequate tensile strength, and foams of higher densities were unnecessarily strong.

The 64 kg/m<sup>3</sup> (4 lb/ft<sup>3</sup>) foam was shown to easily survive the anticipated shuttle ascent, orbit and entry environments, while maintaining good insulating characteristics. As expected, the thermal conductivity varied with temperature, but did not exceed 8.3 watts/m·°K (1.2 Btu-in./hr-ft<sup>2</sup>-°F) up to 640°K (690°F).

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\*Monsanto Company RI-7271-01 foamable polyimide powder. This polyimide has a linear, completely aromatic structure.

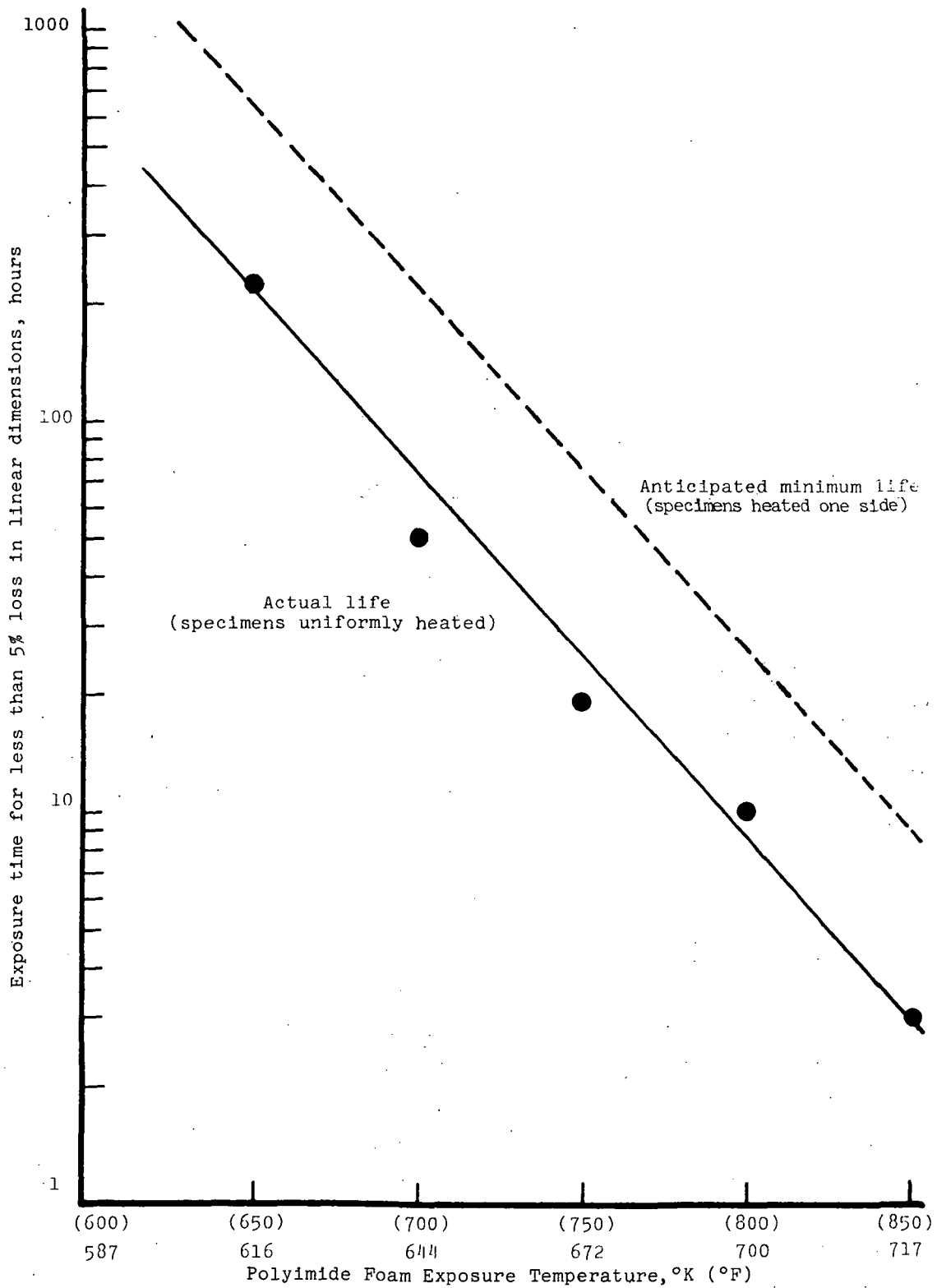


Figure 1. Anticipated Usable Life of Polyimide Foam in Shuttle Application as a Function of Use Temperature.

Costs for the optimized foam were estimated for three volumes of production. None of these three levels was sufficient to significantly reduce either the material or processing costs because both are in the development stage. The excellent demonstrated performance of the polyimide foam for the shuttle application, however, justifies its present cost. It is expected that the polyimide foam can be used with a high degree of confidence in regions of the shuttle where temperatures will not exceed 670°K (750°F).

## INTRODUCTION

A large part of the upper surface of the space shuttle is expected to be exposed to temperatures of no more than 700°K (800°F). There was evidence indicating that low density (32-138 kg/m<sup>3</sup>; 2-8 lb/ft<sup>3</sup>) polyimide foams such as those developed under contract NAS9-12246 by Monsanto Research Corporation could withstand this temperature for limited periods of time without significant degradation. It was anticipated, then, that if the polyimide foam could withstand the shuttle launch, orbit, and entry flight environments, it should have excellent potential as a reusable upper surface heat shield. The requirements were that it be thermally efficient, have adequate mechanical properties, and have a high performance/cost ratio.

The polyimide foam to be evaluated was based on a low density (8 kg/m<sup>3</sup>; 0.5 lb/ft<sup>3</sup>) polyimide foam molding compound. Means had been developed for fabricating this compound into panels and structures of various densities. This molding compound is shown in Figure 2, and a 2.5 cm (1 inch) thick molded panel is shown in Figure 3.

An objective of this project was to determine the minimum density of the polyimide foam which could be considered to have reuse capabilities in the lower temperature areas of the upper surface of the space shuttle. The limit of this thermal capability was to be defined with respect to the space shuttle application without any major modification to the chemistry of the system. It was necessary that the polyimide foam not restrict heat shield panel size or contribute to heat shield design problems.

The experimental program was to include: (1) an initial foam screening and selection, (2) a determination of the acceptability of the foams in the potential space shuttle environment, (3) a determination of the maximum cyclic reuse temperature for the foam, (4) a definition of the reuse potential and general acceptability of the best candidate foam system, and (5) a cost analysis of the best foam system.





Figure 2. Polyimide Molding Compound ( $8 \text{ kg/m}^3$ ,  $0.5 \text{ lb/ft}^3$ )  
Used to Prepare Thermally Resistant Panels



Figure 3. Molded Polyimide Foam Panels ( $64 \text{ kg/m}^3$ ,  $4 \text{ lb/ft}^3$ )  
That is Resistant to Anticipated Shuttle Environments

The primary independent variables explored in the polyimide foam system were density, and post-curing conditions. The dependent variables examined were: thermal stability; thermal conductivity; resistance to thermal cycling; compressive, flexural, impact and shear strength; low temperature impact strength; high temperature creep; resistance to severe acoustic exposure; and resistance to moisture.

### EXPERIMENTAL DISCUSSION

Polyimide foams ranging in density from 32 to 128 kg/m<sup>3</sup> (2-8 lb/cu ft) provided the basis for this experimental program. All foams were prepared from the same starting materials and were converted in an identical manner. It had been anticipated that post-curing would be necessary to achieve the thermal performance required for the space shuttle application. Accordingly, post-curing techniques were examined and defined to maximize the utility of the polyimide foam.

The experimental results will be discussed in five categories:

- (1) Preparation of the polyimide foam,
- (2) Thermal characteristics of polyimide foam,
- (3) Mechanical characteristics of the polyimide foam,
- (4) Structural properties and moisture resistance of the polyimide foam, and
- (5) Cost analysis of the polyimide foam in the form of thermal protective panels.

#### Preparation of the Polyimide Foam

The preparation of useful polyimide foam panels requires four steps: (1) foaming, (2) molding, (3) post-curing, and (4) bonding to the desired surface with an adhesive.

The foaming and molding procedures were largely developed under NASA contract NAS9-12246, and are described in detail in Appendix A. The post-curing cycles and the effect of this post-curing on dimensional and weight stability were determined during the present investigation.

Preparation of the polyimide foam molding compound. The starting point for the polyimide foam is Monsanto Company's RI-7271-01 foamable polyimide powder. This foamable powder is spread thinly over the bottom of a baking pan and heated for approximately one-half hour at 448°K (347°F). This step causes

the powder to foam to a low density, friable structure. This structure is then converted into a resilient and tough foam by additional curing at 573°K (572°F) for about one-half hour. This very irregular, but strong foam has a density of about 10 kg/m<sup>3</sup> (0.6 lb/cu ft).

Since this foam cannot be formed into shape by confining it in a mold, and since it is very heterogeneous, it must be further processed to be useful. This is done by shredding the foam with a high speed grinder which yields a bulky, light weight, finely divided material (shown in Figure 2) having a density of 8 kg/m<sup>3</sup> (0.5 lb/cu ft). This is the polyimide foam molding compound which can be densified and fused at elevated temperatures.

Molding procedure. The molding procedure is described in detail in Appendix A. Briefly, it involves pouring the shredded polyimide molding compound into a steel mold, compressing it to the desired final dimensions, and fusing it by heating to 600°K (625°F) for several hours. The part is then cooled and removed from the mold.

Post-curing procedure for the polyimide foam. The foam up to this point has been cured for about one-half hour at 573°K (572°F) and about three hours at 600°K (620°F). It would be expected, therefore, that foams made with these cure conditions would be stable over very long periods of time at temperatures up to 600°K (625°F). That is, they should retain their mechanical and thermal characteristics, as well as dimensional stability with respect to both linear dimensions and weight over long periods, i.e. many hundreds of hours.

It was expected that heating the foams above 600°K (620°F) would result in some shrinkage, weight loss, and stiffening. This would result from the more complete imidization of the structure forced by the higher temperatures. It was expected that post-curing to higher temperatures would essentially complete any changes. Therefore, while initial properties would be different, little change in these properties would occur with time at temperatures below the post-curing temperature.

The post-curing schedule established for larger specimens [i.e., 38 cm x 38 cm x 2.5 cm (15 in. x 15 in. x 1 in.)] was: 64 hours at 644°K (700°F), followed by 16 hours at 670°K (750°F), followed by 2 hours at 700°K (800°F). This post-curing was done with the specimen out of the mold in a well-vented oven. About a 10% decrease in volume and weight occurs during this post-curing. The foam also softens in the early stages of the post-cure and sags if not supported.

With smaller specimens (7 cm x 7 cm x 2.5 cm; 3 in. x 3 in. x 1 in.) it was shown that a reasonable post-cure was 16 hours at 700°K (800°F). An additional post-curing to 728°K (850°F) for 4 hours was also possible, but usually not necessary.

In establishing the above optimum post-cure cycle, the weight and dimensional losses of the polyimide foams of various densities were first determined as a function of temperature. The thermally exposed foams were then evaluated qualitatively to determine whether their other physical properties (primarily mechanical) were adequate.

The effect of heating the unpostcured polyimide foam from 67°K (121°F) below the cure temperature to 44°K (79°F) above the cure temperature on weight and dimensions is illustrated in Table 1. Very minor losses in weight and thickness and no changes in length or width occurred at or below a temperature of 587°K (597°F). The more significant weight and dimensional losses occurred above 644°K (700°F). The dimensional losses were higher for foams of lower density, indicating that the lower density foams tend to soften and collapse much more readily than the higher density foams in the range from 32 to 128 kg/m<sup>3</sup> (2 to 8 lb/ft<sup>3</sup>). This examination was conducted on specimens measuring 10 cm x 10 cm x 2.5 cm (4 in. x 4 in. x 1 in.).

The effect of post-curing in increments is shown in Table 2. No major weight losses occurred until around 700°K (800°F).

The effect of post-curing the polyimide foams at 700°K (800°F) for 16 hours on subsequent weight and dimensional changes at lower temperatures is given in Table 3. As expected, changes in weight and shrinkage were reduced at this temperature.

Adhesive bonding of foam to aluminum. Several adhesives were used to bond the post-cured polyimide foam to aluminum. These included silicone (General Electric 1200), epoxy (Epon 828:DETA, 88:12) and Eastman 910®.

The epoxy adhesive system was generally used on specimens prepared for the testing reported here. The only problem with the epoxy was that it tended to wick into the structure of the post-cured polyimide foam, thus reducing the bonding area.

Even though there seemed to be no particular problem using either the epoxy or the silicone systems, it is recommended that a silicone adhesive be used. The higher viscosity of the uncured silicone tends to prevent it from wicking into the polyimide foam and provides for a better bonding surface. The silicone is also somewhat more elastomeric thus providing a transition for differential expansions between the polyimide foam and the aluminum.

Table 1

EFFECT OF TEMPERATURE ON THE WEIGHT AND DIMENSIONAL STABILITY  
OF POLYIMIDE FOAMS<sup>(a)</sup> OF VARIOUS DENSITIES

Approximate Foam Density, as Molded		Weight Loss, %	Thickness Reduction, %	Length & Width Reduction, %
Original Density, kg/m <sup>3</sup>	(lb/ft <sup>3</sup> )			

533°K (500°F), 16 Hours

32	(2)	0.3	0.3	0
48	(3)	0.2	0.3	0
64	(4)	0.3	0.3	0
80	(5)	0.1	0.4	0
96	(6)	0.3	0.3	0
128	(8)	0.2	0.4	0

587°K (600°F), 16 Hours

32	(2)	0.8	1.1	0
48	(3)	0.8	1.0	0
64	(4)	0.9	0.0	0
80	(5)	0.5	0.9	0
96	(6)	0.7	0.7	0
124	(8)	0.6	0.2	0

616°K (650°F), 16 Hours

32	(2)	2.0	7.5	5
48	(3)	2.1	6.3	4.5
64	(4)	1.9	3.3	3
80	(5)	1.7	3.7	2
96	(6)	1.7	3.5	2
128	(8)	1.7	2.4	2.5

644°K (700°F), 16 Hours

32	(2)	2.9	8.7	7
48	(3)	3.0	6.5	5
64	(4)	2.9	3.7	4
80	(5)	2.7	3.9	3
96	(6)	2.7	3.7	3
128	(8)	2.8	2.7	3

(a) molded at 600°K (620°F), no postcure

Table 2

EFFECT OF INCREMENTALLY INCREASED TEMPERATURE ON THE WEIGHT AND DIMENSIONAL STABILITY OF POLYIMIDE FOAMS (a)

		<u>Total Accumulative Change</u>			
<u>Density Measured,</u> <u>kg/m<sup>3</sup> (lb/ft<sup>3</sup>)</u>		<u>Density Increase,</u> <u>%</u>	<u>Weight Loss,</u> <u>%</u>	<u>Thickness</u> <u>Reduction,</u> <u>%</u>	<u>Length &amp; Width</u> <u>Reduction, %</u>
<u>After initial 16 hours at 533°K (500°F)</u>					
48	(3.0)	0	<0.2	0.3	0
69	(4.3)	0	0.3	0.2	0
86	(5.4)	0	0.1	0.4	0
<u>Additional 16 hours at 587°K (600°F)</u>					
48	(3.0)	0	<0.2	1.3	0
69	(4.3)	0	0.4	0.2	0
86	(5.4)	0	0.1	0.9	0
<u>Additional 16 hours at 616°K (650°F)</u>					
56	(3.5)	17	1.6	6.5	4
75	(4.7)	7	1.5	3.0	3
90	(5.6)	6	1.3	3.2	2
<u>Additional 16 hours at 644°K (700°F)</u>					
56	(3.5)	17	3.0	7.0	5
75	(4.7)	7	3.0	3.6	4
91	(5.7)	8	2.6	3.9	2
<u>Additional 4 hours at 672°K (750°F)</u>					
58	(3.6)	20	3.4	7.7	6
77	(4.8)	9	3.5	4.2	4
93	(5.8)	9	3.2	4.2	3
<u>Additional 16 Hours at 700°K (800°F)</u>					
56	(3.5)	17	8.5	9.2	7
77	(4.8)	9	8.2	5.8	6
91	(5.7)	8	7.4	5.6	4
<u>Additional 16 hours at 700°K (800°F)</u>					
56	(3.5)	15	20	13	10
74	(4.6)	5	24	10	11
90	(5.6)	5	21	9	8
<u>Additional 16 hours at 700°K (800°F)</u>					
50	(3.1)	3	46	22	18
64	(4.0)	-9	55	21	22
82	(5.1)	-4	45	17	17

(a) molded at 600°K (620°F)

Table 3

SHORT TERM THERMAL STABILITY OF POSTCURED POLYIMIDE FOAMS (a) OF VARIOUS DENSITIES

Initial Postcured Foam Density kg/m <sup>3</sup> (lb/ft <sup>3</sup> )	After 16 Hours at 644°K (700°F) Exposure			Average Length & Width Decrease, %
	Foam Density After Exposure kg/m <sup>3</sup> (lb/ft <sup>3</sup> )	Weight Loss, %	Thickness Decrease, %	
55.5 (3.47)	55.5 (3.47)	2	0.9	0.5
71.5 (4.47)	70.9 (4.43)	2	0.5	0.2
85.9 (5.37)	85.9 (5.37)	1	0.5	0.3

(a) Foams were molded at 600°K (620°F) and postcured at 700°K (800°F) for 16 hours. Sample dimensions were approximately 5 cm x 5 cm x 2.5 cm (2 in. x 2 in. x 1 in.).

Testing of post-cured  $64 \text{ kg/m}^3$  ( $4 \text{ lb/ft}^2$ ) foams bonded with the three adhesives showed that they were all good from  $144^\circ\text{K}$  ( $-200^\circ\text{F}$ ) to  $394^\circ\text{K}$  ( $250^\circ\text{F}$ ) in a partial vacuum (approximately 18 torr). After heating the bonded specimens at  $700^\circ\text{K}$  ( $800^\circ\text{F}$ ) for 30 minutes in air, however, only the silicone-bonded foam retained a reasonable degree of adhesion. In these cases the adhesives also were heated to  $700^\circ\text{K}$  ( $800^\circ\text{F}$ ), which is not anticipated in the shuttle application.

### Thermal Characteristics of the Polyimide Foam

Dominant in demonstrating the utility of the foam for the shuttle application are its thermal characteristics. These specifically include its resistance to elevated temperatures near  $700^\circ\text{K}$  ( $800^\circ\text{F}$ ), its insulating characteristics in terms of thermal conductivity at these elevated temperatures, and its ability to withstand thermal cycling between these elevated temperatures and near liquid nitrogen temperatures.

#### Resistance of the polyimide foam to elevated temperatures relative to the foam density and environment during heating.

The weight loss of unpostcured polyimide foams (molded at  $600^\circ\text{K}$ ;  $620^\circ\text{F}$ ) was determined as a function of temperature up to  $700^\circ\text{K}$  ( $800^\circ\text{F}$ ) and then as a function of time in air. Foams of three densities including 48, 64, and  $90 \text{ kg/m}^3$  (3, 4 and  $5 \text{ lb/ft}^3$ ) were examined.

The heating rate from ambient to  $700^\circ\text{K}$  ( $800^\circ\text{F}$ ) was  $6^\circ\text{K}$  ( $11^\circ\text{F}$ ) per minute. It was then held at  $700^\circ\text{K}$  ( $800^\circ\text{F}$ ) for 120 minutes. The results of this thermogravimetric analysis during initial heating and isothermal exposure are illustrated in Figure 4. The percent weight loss for all three foams was nearly identical, as might be expected. About a 2% weight loss was attributed to moisture. The final total weight loss was 6%. Accordingly, a 4% weight loss could be attributed to the polyimide itself. This was to be expected since further imidization should occur between the curing temperature of  $600^\circ\text{K}$  and its exposure temperature of  $700^\circ\text{K}$  ( $800^\circ\text{F}$ ).

The effects of various atmospheres on the weight loss due to temperature were examined on a  $64 \text{ kg/m}^3$  ( $4 \text{ lb/ft}^3$ ) unpostcured polyimide foam. The atmospheres investigated included air, a good vacuum of less than 1 torr, a partial vacuum of 18 torr (suggested for space simulation), and helium.



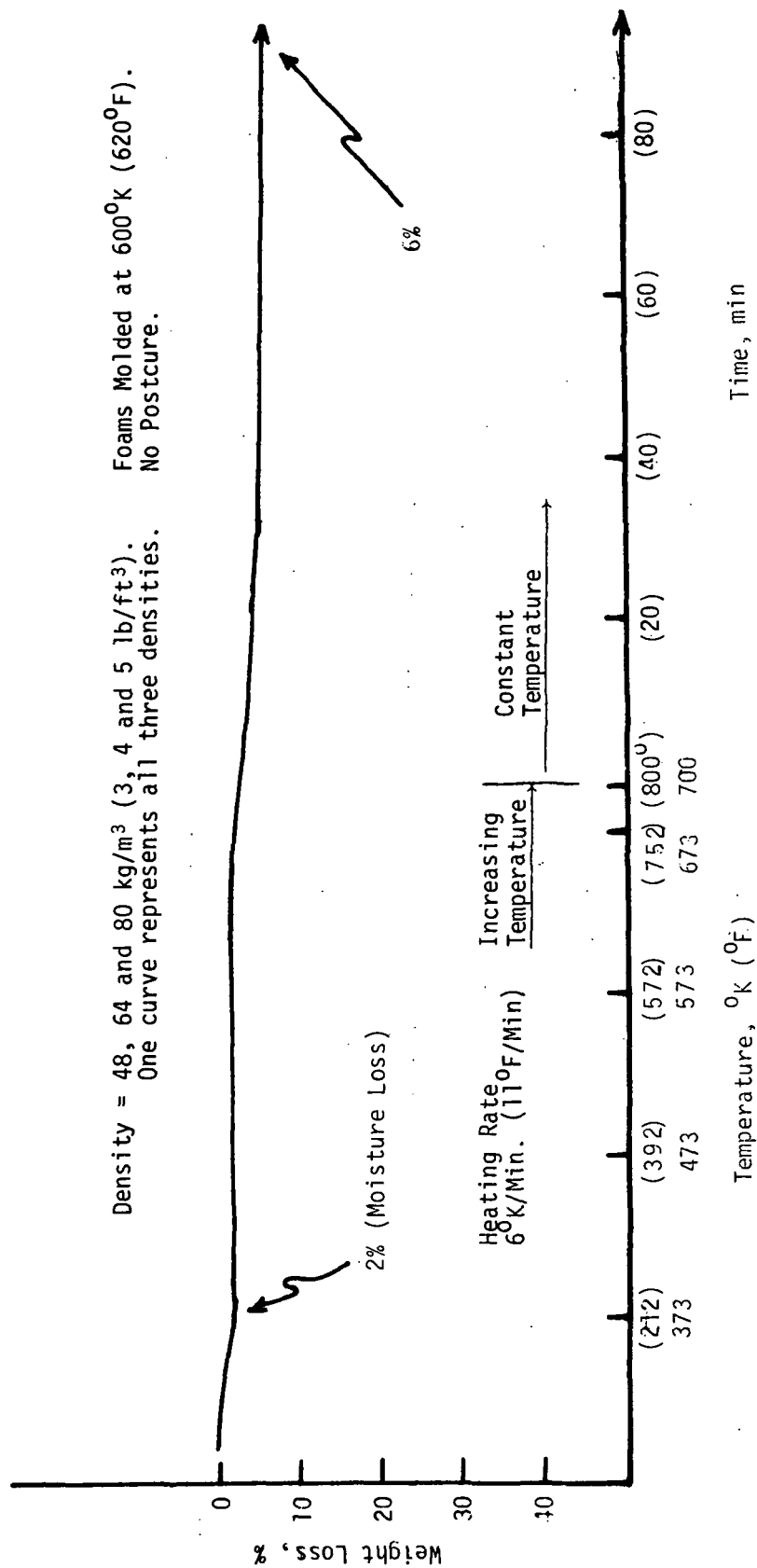


Figure 4. Thermogravimetric Analysis of 48, 64 and 80 kg/m<sup>3</sup> (3, 4 and 5 lb/ft<sup>3</sup>) Polyimide Foams Heated to 700°K (800°F) and Then Held Isothermally for 120 Minutes.

Foams were heated to 1270°K (1830°F) in a thermogravimetric analyzer using a heating rate of 3°K (5°F) per minute. The results are given in Table 4 and Figures 5 and 6. Interestingly, a weight loss of around 20% occurred between 800-850°K (980-1070°F), independent of the environment. However, total weight loss differed greatly at 1000°K, 1341°F (see Figure 5). Air was the most detrimental environment while helium and the 18-torr vacuum had about the same effect and were the least severe. There is no doubt that oxygen is detrimental to the life of the polyimide foam at elevated temperatures.

Thermal conductivity of the polyimide foams. - The primary purpose of the polyimide foam in the shuttle application is to provide a thermal barrier between the external environment and the aluminum skin. Accordingly, the thermal conductivity, especially as a function of temperature, is a most important parameter. Thermal conductivity was measured on the polyimide foams first using a simplified screening test and finally with a guarded hot plate, according to the ASTM C-177 procedure.

The thermal conductivity screening test was conducted using the unguarded hot plate arrangement shown in Figure 7. Here, the top side of the foam is contacted with a heated surface (hot plate) at a temperature of 600°K (620°F) and a 1/4-inch thick (0.635 cm) aluminum disk contacts the bottom surface. The foam specimen is allowed to soak at test temperature until an equilibrium is reached. The differential temperature across the foam and the mean temperature (which is the average of the top and bottom temperatures) are then used to calculate a thermal conductivity value. In this manner, foams having densities from 32 to 160 kg/m<sup>3</sup> (2-10 lb/ft<sup>3</sup>) were screened for thermal conductivity up to mean temperatures of 470°K (390°F). The results, shown in Table 5 and Figure 8, indicate, as expected, that thermal conductivity increases with temperature and density. Accordingly, the foam with the minimum thermal product (p·k) was that with the lowest density. Therefore, the limiting factor was not the thermal conductivity or the density, but the mechanical properties of the materials at the lower densities. These results led us to eliminate from consideration the higher density foams and examine only those having densities of 48, 64, 80 and 96 kg/m<sup>3</sup> (3, 4, 5 and 6 lb/ft<sup>3</sup>).

Having established that the lower density polyimide foams were the best in terms of thermal conductivity using the screening method, standard tests were conducted according to ASTM C-177 procedure on 48 and 96 kg/m<sup>3</sup> (3 and 6 lb/ft<sup>3</sup>) uncured foams and a 64 kg/m<sup>3</sup> (4 lb/ft<sup>3</sup>) postcured foam. This test method uses 20-cm diameter, 2.5-cm thick disks (8 in. diameter

TEMPERATURES AND ENVIRONMENTS ASSOCIATED WITH GIVEN WEIGHT LOSSES  
FOR POLYIMIDE FOAM<sup>(a)</sup>

Weight Loss, %	Temperature at Indicated Weight Loss (b)							
	Air		18 Torr Vacuum		<1 Torr Vacuum		Helium	
	°K	(°F)	°K	(°F)	°K	(°F)	°K	(°F)
0	613	(644)	583	(590)	473	(392)	593	(608)
1	673	(752)	653	(716)	533	(500)	653	(716)
10	803	(986)	823	(1022)	813	(1004)	813	(1004)
20	813	(1004)	853	(1076)	845	(1062)	853	(1076)

(a) molded at 600°K, no postcure, density = 64 kg/m<sup>3</sup> (4 lb/ft<sup>3</sup>).

(b) applied heating rate =  $3^{\circ}\text{K}/\text{min}$  ( $5.4^{\circ}\text{F}/\text{min}$ ) in TGA unit.

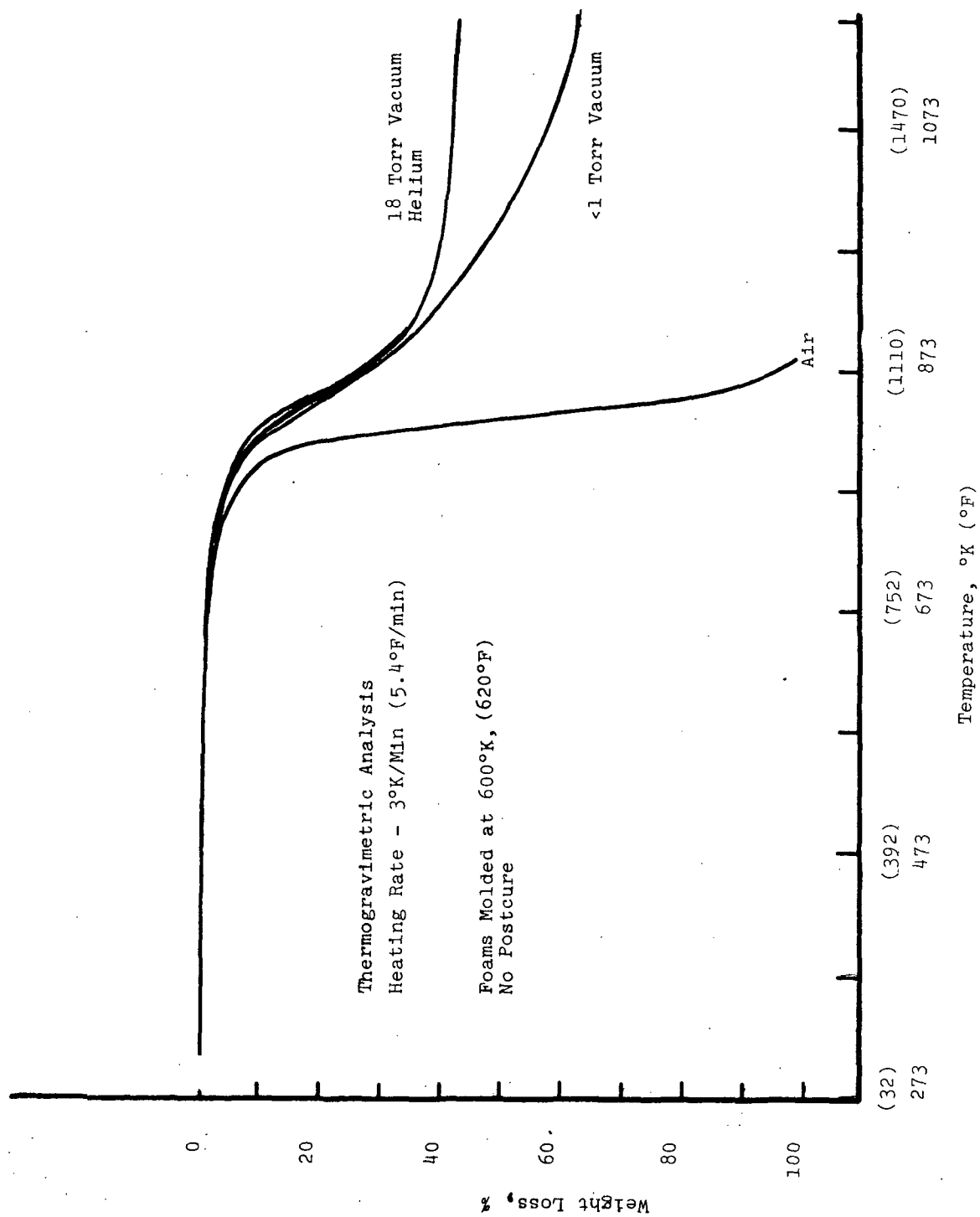


Figure 5. Weight Loss of 64 kg/m<sup>3</sup> (4 lb/ft<sup>3</sup>) Polyimide Foam in Various Environments up to 1273°K (1830°F).

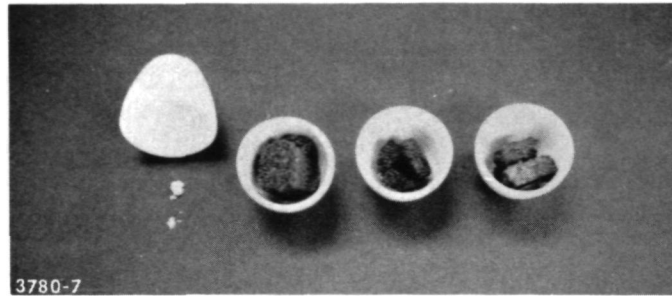


Figure 6. Material Remaining After Heating of Polyimide Foams ( $64 \text{ kg/m}^3$ ,  $4 \text{ lb/ft}^3$  density) to  $1273^\circ\text{K}$  ( $1830^\circ\text{F}$ ) in Various Environments. From left to right: Air; 18 Torr Vacuum, <1 Torr Vacuum, Helium

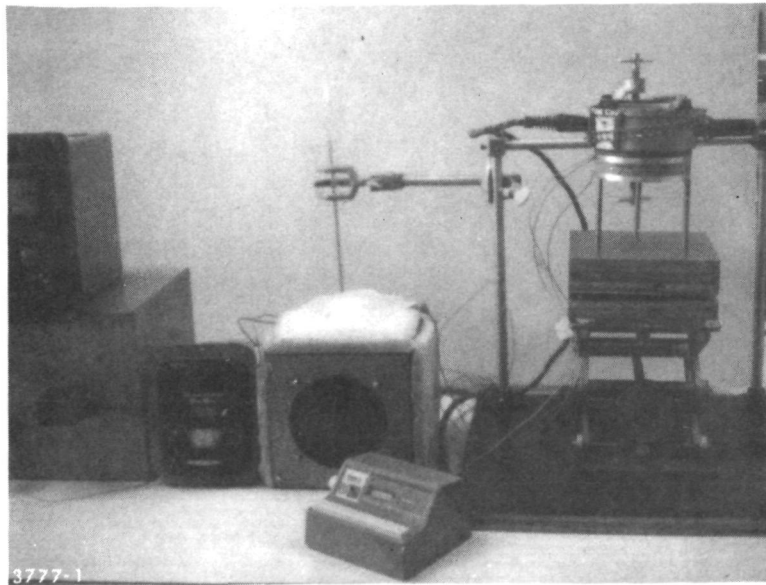


Figure 7. Apparatus for Thermal Conductivity Screening Tests for Polyimide Foams to  $600^\circ\text{K}$  ( $620^\circ\text{F}$ )

Table 5

THERMAL CONDUCTIVITY OF POLYIMIDE FOAM<sup>(a)</sup> AS A FUNCTION OF DENSITY  
USING SCREENING TEST

Density ( $\rho$ )		Thermal Conductivity <sup>(b)</sup> (K)		
<u>kg/m<sup>3</sup></u>	<u>lb/ft<sup>3</sup></u>	<u>Watts/m·°K</u>	<u>Btu·in./h ft<sup>2</sup>·°F</u>	<u>(<math>\rho \cdot K</math>)</u>
32	2	.0379	.263	1.21
48	3	.0385	.267	1.85
64	4	.0389	.270	2.49
80	5	.0394	.273	3.15
96	6	.0398	.276	3.82
128	8	.0408	.283	5.22
160	10	.0418	.290	6.69

(a) molded at 600°K (620°F), no postcure.

(b) K measured at mean temperature approximately 467°K (381°F)  
 upper plate temperature = 536°K (505°F); lower plate  
 temperature = 297°K (255°F).

Method: Unguarded hot plate, equilibrium technique of Lees,  
 described in Jerrard and McNeill, "Theoretical and  
 Experimental Physics," Chapman and Hall, London,  
 1960.

Estimated error:  $\pm 5\%$ . Reported data obtained from linear  
 curve fit over density range. Sample size:  
 10.2 cm (4 in.) diameter, 0.64 cm (0.25 in.) thick.

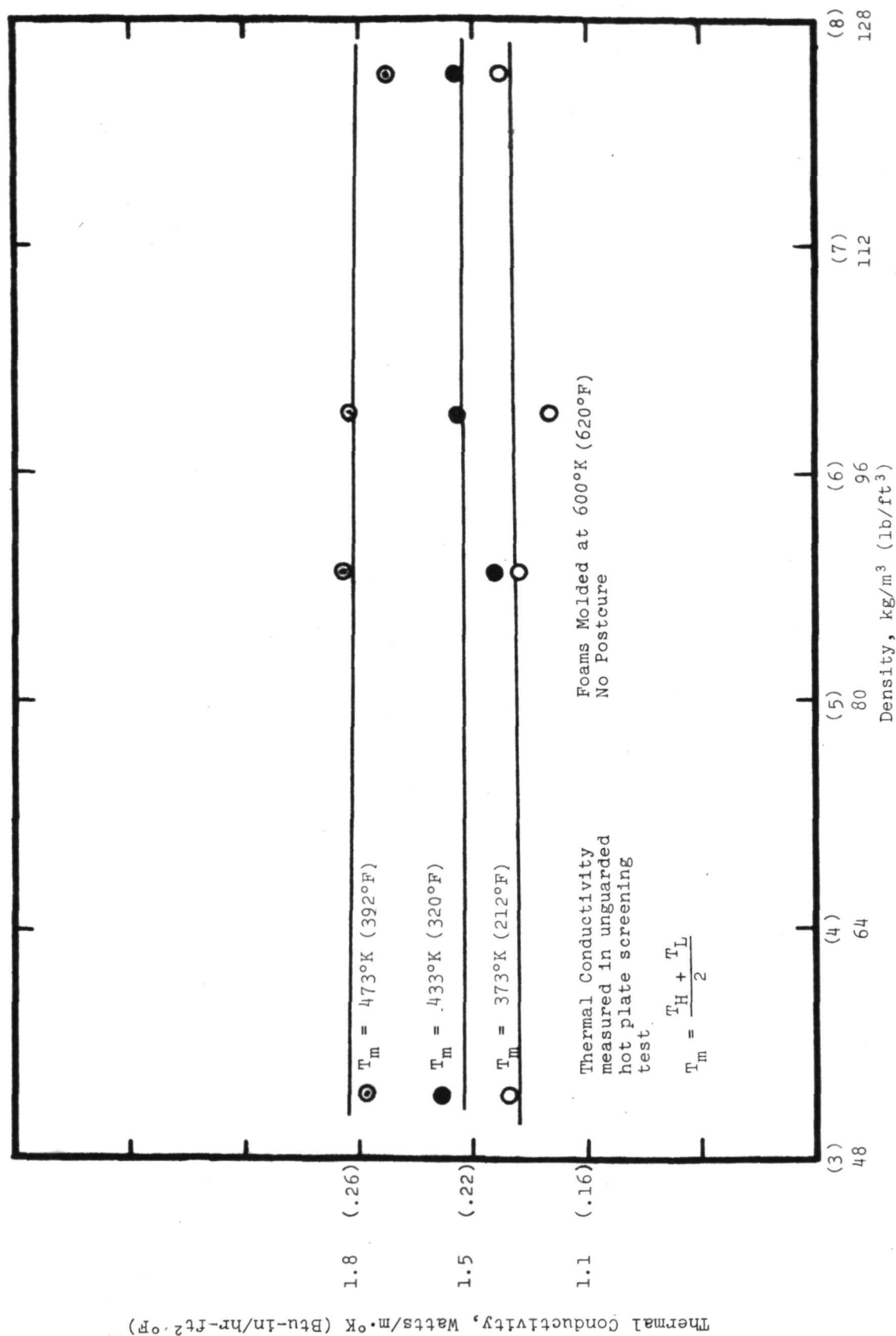


Figure 8. Thermal Conductivity - Density Relationship at Several Mean Temperatures for Polyimide Foams.

x 1 in. thick) in a guarded hot plate arrangement. A few of these test disks are shown in Figure 9. In contrast to the screening test, both sides of the test specimens are heated (a matched pair is used), which allows the mean temperature to be brought closer to the desired limit without causing degradation on the hot side by overheating.

Figure 10 illustrates the resultant thermal conductivities as a function of temperature for the three foams tested (two uncured foams at two densities and a postcured foam at a density of  $64 \text{ kg/m}^3$ ;  $4 \text{ lb/ft}^3$ ). The post-cure on this foam was 64 hours at  $644^\circ\text{K}$  ( $700^\circ\text{F}$ ) followed by 16 hours at  $670^\circ\text{K}$  ( $750^\circ\text{F}$ ), followed by 2 hours at  $700^\circ\text{K}$  ( $800^\circ\text{F}$ ). This testing was done by McDonnell Aircraft (MCAIR) in St. Louis; the detailed test results are given in Appendix B.

Illustrated in Figure 10 is the fact that the lower density foam has a lower thermal conductivity at the lower temperatures. However, this thermal conductivity increased more rapidly than that of the higher density ( $96 \text{ kg/m}^3$ ;  $6 \text{ lb/ft}^3$ ) foam up to about  $570^\circ\text{K}$  ( $567^\circ\text{F}$ ). Thus, in this range, the thermal conductivity of the air dominated.

Significantly, the thermal conductivity of the post-cured  $64 \text{ kg/m}^3$  ( $4 \text{ lb/ft}^3$ ) foam was lower originally (at low temperature). It increased along the lines of the  $48 \text{ kg/m}^3$  ( $3 \text{ lb/ft}^3$ ) foam to  $477^\circ\text{K}$  ( $400^\circ\text{F}$ ) and then decreased. An important observation was that the uncured foam did shrink some during the heating, while the post-cured foam remained dimensionally stable, even upon some inadvertent heating to near  $755^\circ\text{K}$  ( $900^\circ\text{F}$ ).

Thermal insulating properties of the polyimide foam. - It was important that the thermal conductivity be known as a function of temperature so that NASA could calculate thermal gradients for a particular design configuration and thickness of the polyimide foam. It was also desirable that a rough determination be made experimentally to illustrate the actual thermal insulating properties of the  $64 \text{ kg/m}^3$  ( $4 \text{ lb/ft}^3$ ) foam at a given hot-side temperature.

The thermal insulating properties were determined using both the screening test apparatus (a hot contacting metal surface) and hot forced air. The resultant data on both uncured and post-cured specimens at skin temperatures of about  $600^\circ\text{K}$  ( $620^\circ\text{F}$ ) are shown in Table 6.

The thickness of the foams was 2.3 cm (0.91 in.). When the top surfaces were at  $614^\circ\text{K}$  ( $645^\circ\text{F}$ ) the bottom sides were  $350$  and  $384^\circ\text{K}$  ( $171$  and  $232^\circ\text{F}$ ), respectively, for the uncured and post-cured specimens. The bottom surfaces contacted a 0.16 cm (1/16 in.) aluminum plate which was free to radiate into air at around  $296^\circ\text{K}$  ( $73^\circ\text{F}$ ). Quite similar results were shown when the hot side was



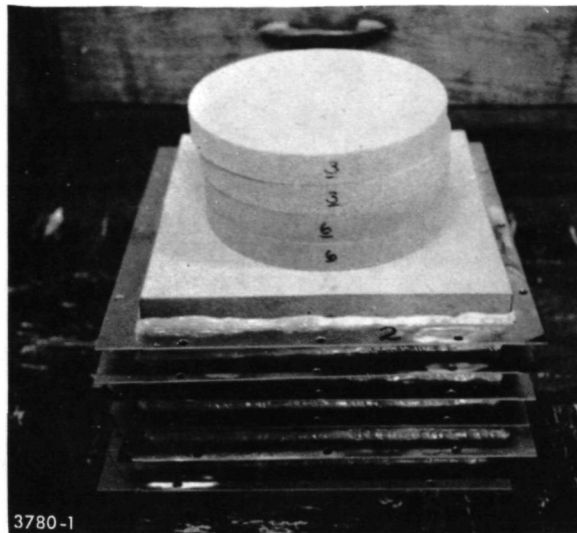


Figure 9. 20.3 cm (8 in.) Diameter Disks for ASTM Guarded Hotplate Thermal Conductivity Measurements and Square Foot Panels Bonded to Aluminum Plate for Acoustic Loading Testing

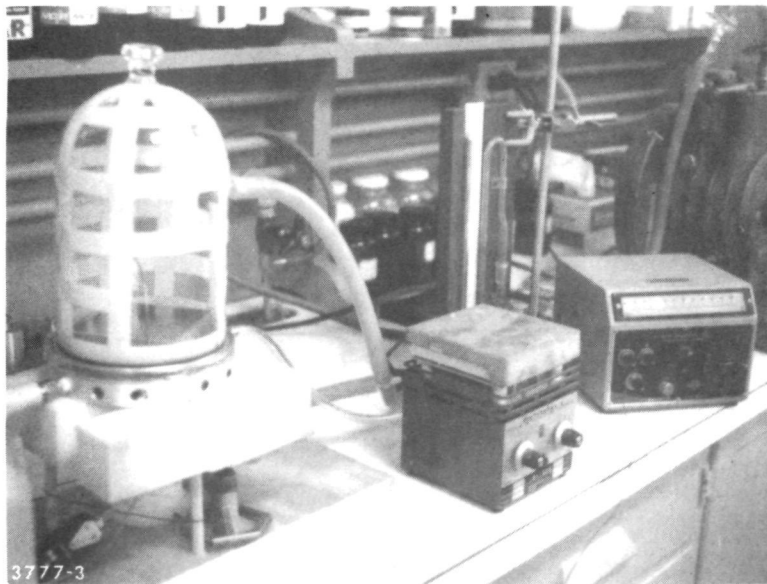


Figure 11. Low and High Temperature Reduced Pressure Apparatus for Environmental Cyclic Testing of Polyimide Foams. Samples 9 cm x 9 cm x 2.5 cm (3.5 in. x 3.5 in. x 1 in.)

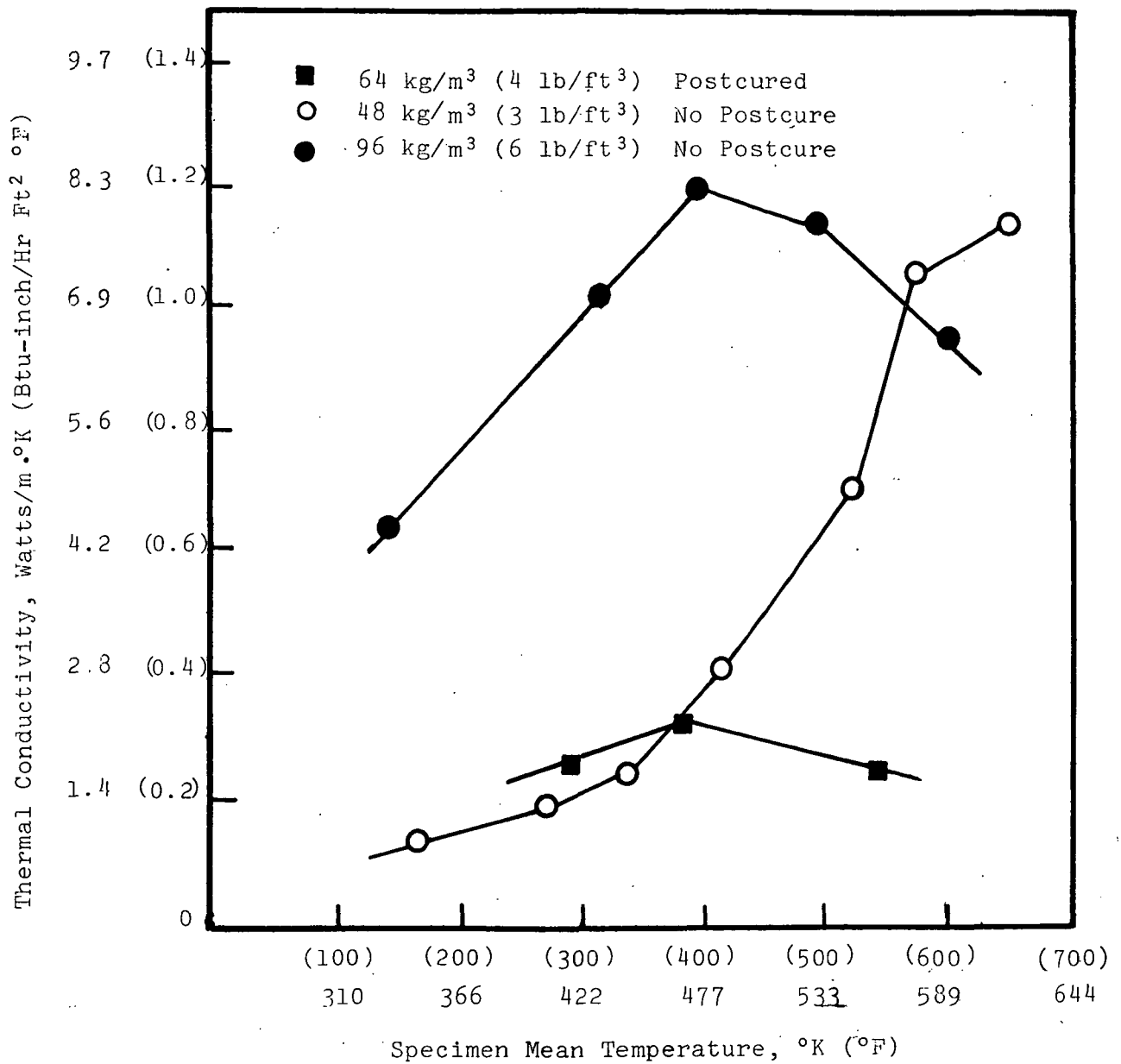


Figure 10. Thermal Conductivity of Polyimide Foams as a Function of Density, Mean Temperature and Cure

Table 6

THERMAL INSULATING PROPERTIES (a) OF 64 kg/m<sup>3</sup> (4 lb/ft<sup>3</sup>) POLYIMIDE FOAMS (b)  
BASED ON MEASURED DIFFERENTIAL TEMPERATURE

Foam Cure	Thick- ness, cm	Metal Surface at 614°K(646°F)		Forced Air at 525°K(572°F)	
		Bottom Surface Temperature	Differential Temperature	Bottom Surface Temperature	Differential Temperature
molded at 600°K (620°F), no postcure	2.3 (0.91 in.)	350°K(171°F)	264°K(471°F)	344°K(160°F)	279°K(412°F)
molded at 600°K (620°F) postcured 700°K(800°F) for 16 hrs	2.3 (0.91 in.)	384°K(232°F)	230°K(410°F)	350°K(171°F)	223°K(401°F)

- (a) Temperature differential across foam with top side heated by contact with hot plate or air stream and bottom contacting 1/16" aluminum sheet in RT air.
- (b) molded at 600°K (620°F) density 64 kg/m<sup>3</sup> (4 lb/cu ft).

heated with forced air across its surface. While the differential across the post-cured foam was less than that across the uncured foam, in all cases a differential of at least 220°K (400°F) was achieved. These measurements were all made at equilibrium, which required hours to achieve (much more than is expected or required for reentry of the space shuttle). Additionally, the back side temperatures were all less than 385°K (230°F), which is well below structural thresholds for the aluminum [expected to be around 450°K (350°F)].

Resistance of the polyimide foam to thermal cycling (orbit simulation). - In the orbiting shuttle environment it is expected that the thermal insulation will be cycled between 144°K (-200°F) and 394°K (250°F) repeatedly. This would occur in a vacuum at approximately 18 torr. Accordingly, both uncured and post-cured polyimide foams ranging in density from 32 to 128 kg/m<sup>3</sup> (2 to 8 lb/ft<sup>3</sup>) were examined under the simulated conditions. Thermal cycles down to as low as 77°K (-321°F) were utilized as well.

The apparatus used for the thermal cycling experiment is shown in Figure 11. It included a heating and cooling plate that was covered with a bell jar. The plate could be cooled rapidly to 144°K (-200°F) by pumping liquid nitrogen through it, and could be cycled to higher temperatures (i.e., 400°K; 261°F) by transferring the plate to a preheated hot plate. The bell jar was used to provide a partial vacuum over the specimens. The specimens were rectangular pieces 9 cm x 9 cm x 2.5 cm (3.5 in. x 3.5 in. x 1 in.) with 0.16 cm thick aluminum sheet bonded with an epoxy adhesive to one surface. Thermocouples were placed at both the top and bottom of the specimen to monitor temperature.

The stability of the polyimide foams to the thermal cycling environment were examined both with the bonded aluminum sheet touching the temperature cycling surface and separately with just the foam contacting the surface.

Foams molded at 600°K (620°F), but uncured, having densities from 32 to 128 kg/m<sup>3</sup> (2 to 8 lb/ft<sup>3</sup>) and post-cured foams in the density range from 56 to 64 kg/m<sup>3</sup> (3.5 to 4 lb/ft<sup>3</sup>) were examined. These foams were post-cured for 16 hours at 700°K (800°F). Upon thermal cycling these foams from 144 to 394°K (-200-250°F) with the bonded aluminum plate either contacting the temperature cycling plate, or away from it, no sign of any degradation, cracking, distortion, or failure was noted. Weights and dimensions of the specimens varied by less than 0.3% from the start to the finish of the thermal cycling. The weight loss was attributed to moisture and exposure to the partial vacuum. The weight was regained when the panels were exposed to ambient conditions for 24 hours.

Since no failures were found to result from thermal cycling from 144°K to 394°K (-200-250°F) and back in an 18 torr vacuum; 2.5 cm cubes of the polyimide foam in densities from 32 to 128 kg/m<sup>3</sup> (2-8 lb/ft<sup>3</sup>) were soaked in liquid nitrogen (77°K; -321°F) until equilibrated, removed, and immediately placed in a vacuum oven at 394°K (250°F). No failure or degradation of any sort was observed in these thermally shocked specimens.

### Mechanical Characteristics of the Polyimide Foam

The mechanical properties of the polyimide foams were examined to provide a basis for selection of the optimum density, to determine variations in properties with density, and to define the nature of the polyimide foam with respect to the requirements for the shuttle application (launch, orbit, and entry). The bulk of the properties were determined at room temperature, but some consideration was also given to sub-ambient and elevated temperatures where the foam must perform, although not under too severe mechanical conditions.

Characteristics of the polyimide foam at room temperature: Properties examined at room temperature included resistance to compression, flexure, impact, severe acoustic forces, and surface pressures. Both uncured and cured foams were examined, with density being the prime variable.

Compressive Strength: Compressive-stress-strain characteristics were determined according to ASTM D-1564 using 5 cm x 5 cm x 2.5 cm (2 in. x 2 in. x 1 in.) specimens. The compressive properties were determined as a function of density from 32 to 128 kg/m<sup>3</sup> (2-8 lb/ft<sup>3</sup>). Strength as a function of density at various degrees of compression are illustrated in Figure 12. As expected, the compressive strengths increased with density in a somewhat geometric manner. The data shown in Figure 12 are for foams that were not cured. The fact that they could be compressed to a level of 80% is a strong indication of their toughness. Significantly, also, all of the specimens recovered to within 5% of their original thickness after being compressed to the 80% level, indicating a good degree of resiliency and ability to withstand compressive loads without failing.

The effect of curing of the polyimide foams at densities of 48 to 64 kg/m<sup>3</sup> (3-4 lb/ft<sup>3</sup>) on compressive properties is illustrated in Figure 13. As expected, the compressive strengths at a given

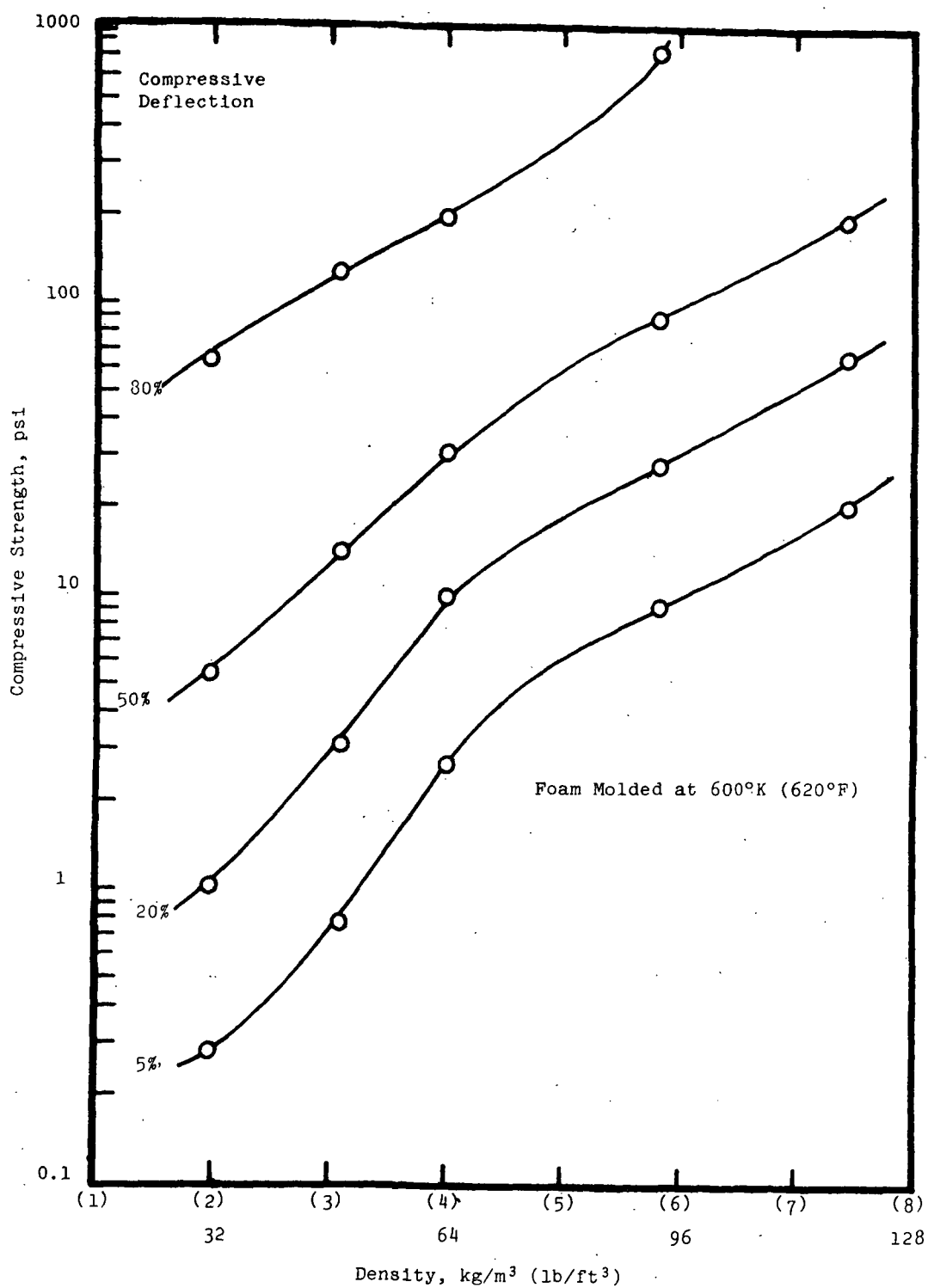


Figure 12. Compressive Strength as a Function of Density for Unpostcured Polyimide Foams at Selected Compressive Deflections.

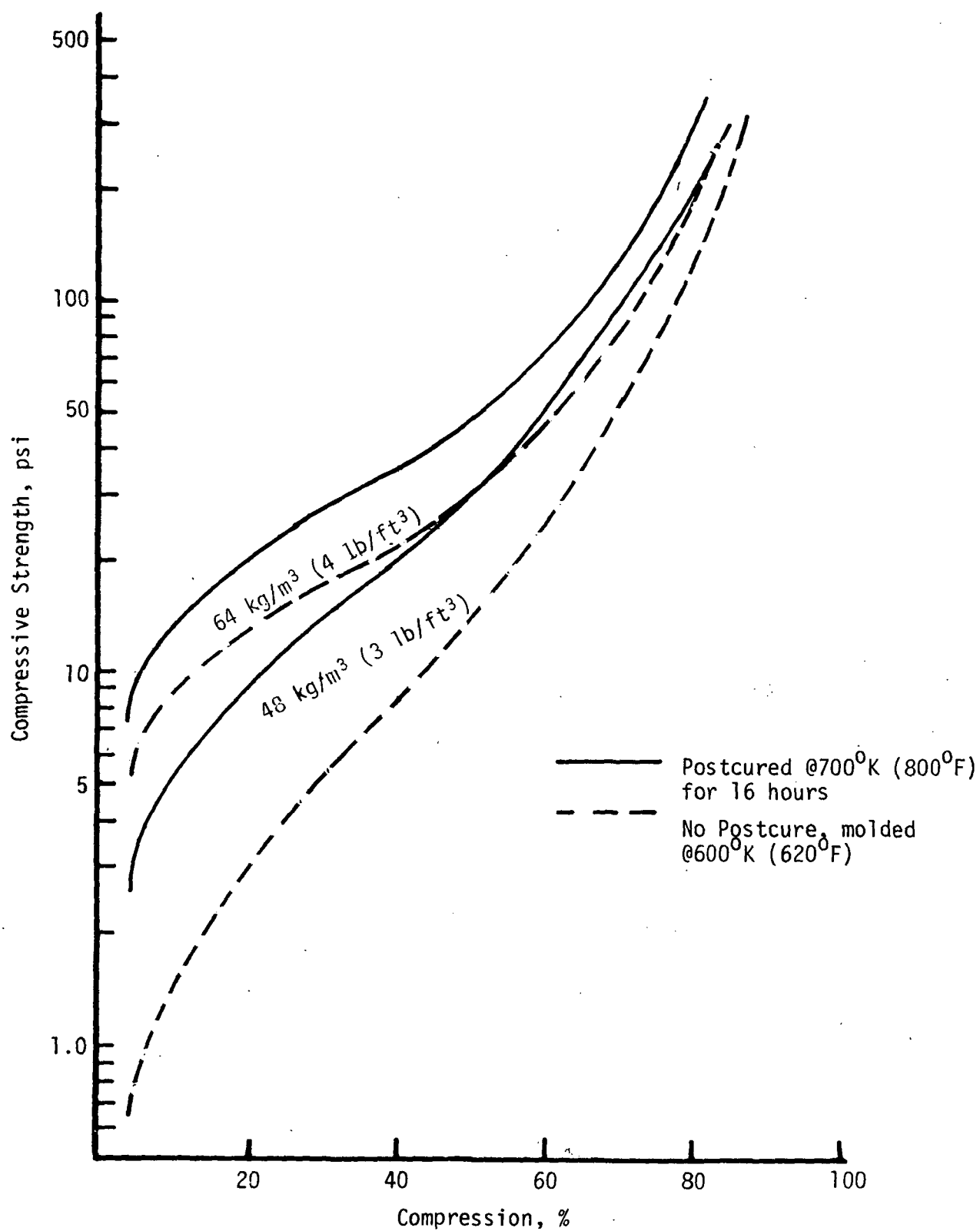


Figure 13. Effect of Postcuring on the Compressive Characteristics of 48 and 64 kg/m<sup>3</sup> (3 and 4 lb/ft<sup>3</sup>) Density Polyimide Foams.

deflection are higher, and some minor amount of resiliency is lost. However, again, even the postcured polyimide foams recovered to within 5% of their original thickness after being compressed 80%.

Flexural Properties: Although the polyimide foam will be subjected to little in the way of flexural loads, especially since it will be backed up with a relatively rigid substrate in the shuttle application, the expected highly desirable and acceptable flexural characteristics of the polyimide foam were quantified. A specimen configuration slightly less than standard was utilized. The test beam was a 20.3 cm (8 in.) long beam having a width of 2.5 cm (1 in.) and a thickness of 2.5 cm (1 in.). The beam span and loading dimensions are shown in Figure 14 for the three-point loading test used. This test subjects the foam to both compressive and tensile stresses. Loads were applied using a method which permitted test to failure.

The results of the flexural tests are shown in Figure 15 for foams of three densities, both as-molded and following a 16-hour, 700°K (800°F) postcure. These data show the expected increases in rigidity as a function of density and the very small changes that occurred in flexural properties as a result of the postcuring. The main difference in the postcured specimens was a slight decrease in ultimate strength (load at failure in bending).

Impact Resistance: Important for the application of the polyimide foam in the shuttle is its resistance to impact from dropping of various items such as hammers, screw drivers, wrenches, etc., and propelled items such as rocks, stones, hail, etc. The test results most indicative of such exposure involved impact by a dropped steel ball. Using this technique, both impact resistance and rebound characteristics can be illustrated. The steel ball used as a 2.2-cm diameter (0.866 in.), 45-gram (0.090 lb) sphere which was dropped 89 cm (34 in.) onto foam specimens that were 2.5 cm (1 in.) thick and 5 cm x 5 cm (2 in. x 2 in.) in cross section. Both rebound height of the sphere and sample behavior were recorded.

The impact resistance of the polyimide foams ranging in densities from 48 to 80 kg/m<sup>3</sup> (3-5 lb/ft<sup>3</sup>) both as-molded and with postcures is given in Table 7. These results show that even at the lowest density only a very slight permanent depression was created, and the steel ball bounced back up to 48% of its drop height.

There was a slight decrease in the impact resistance due to postcuring; this is indicated in Table 7 as a slightly increased permanent depression and slightly lower resilience (percent rebound). Importantly, this surface depression was not only



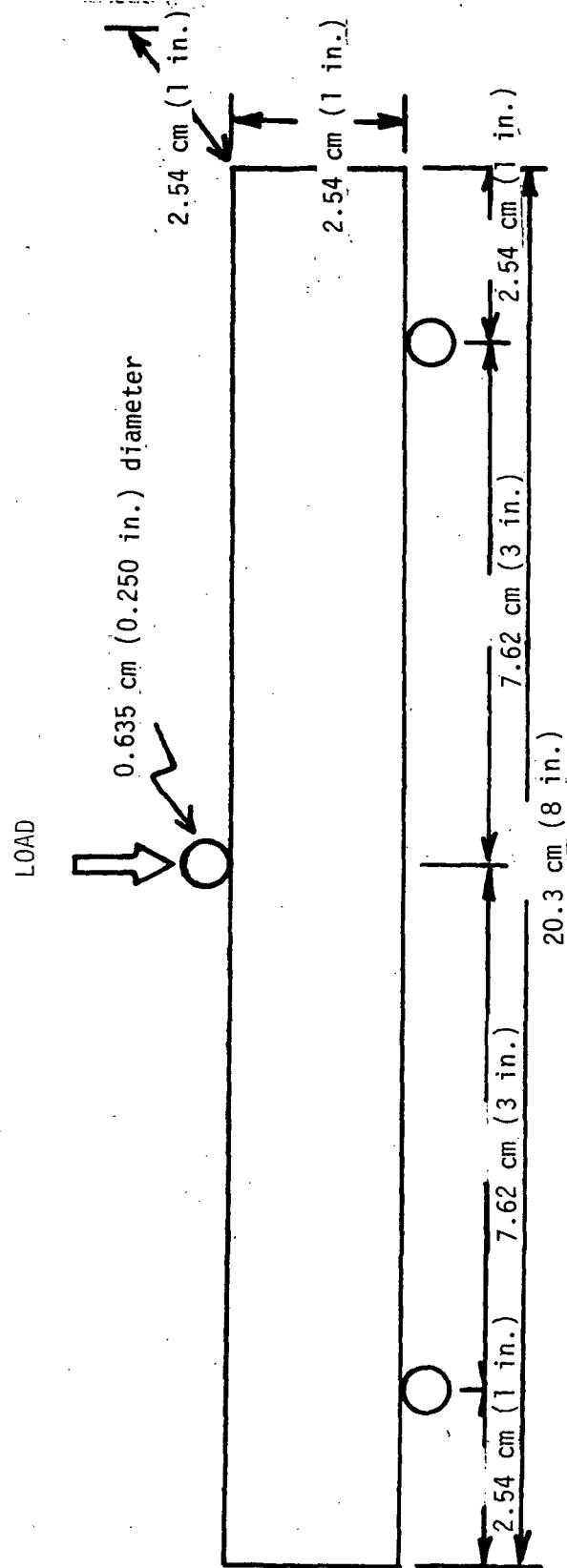


Figure 14. Beam Span and Loading Dimensions for Foam Flexural Test

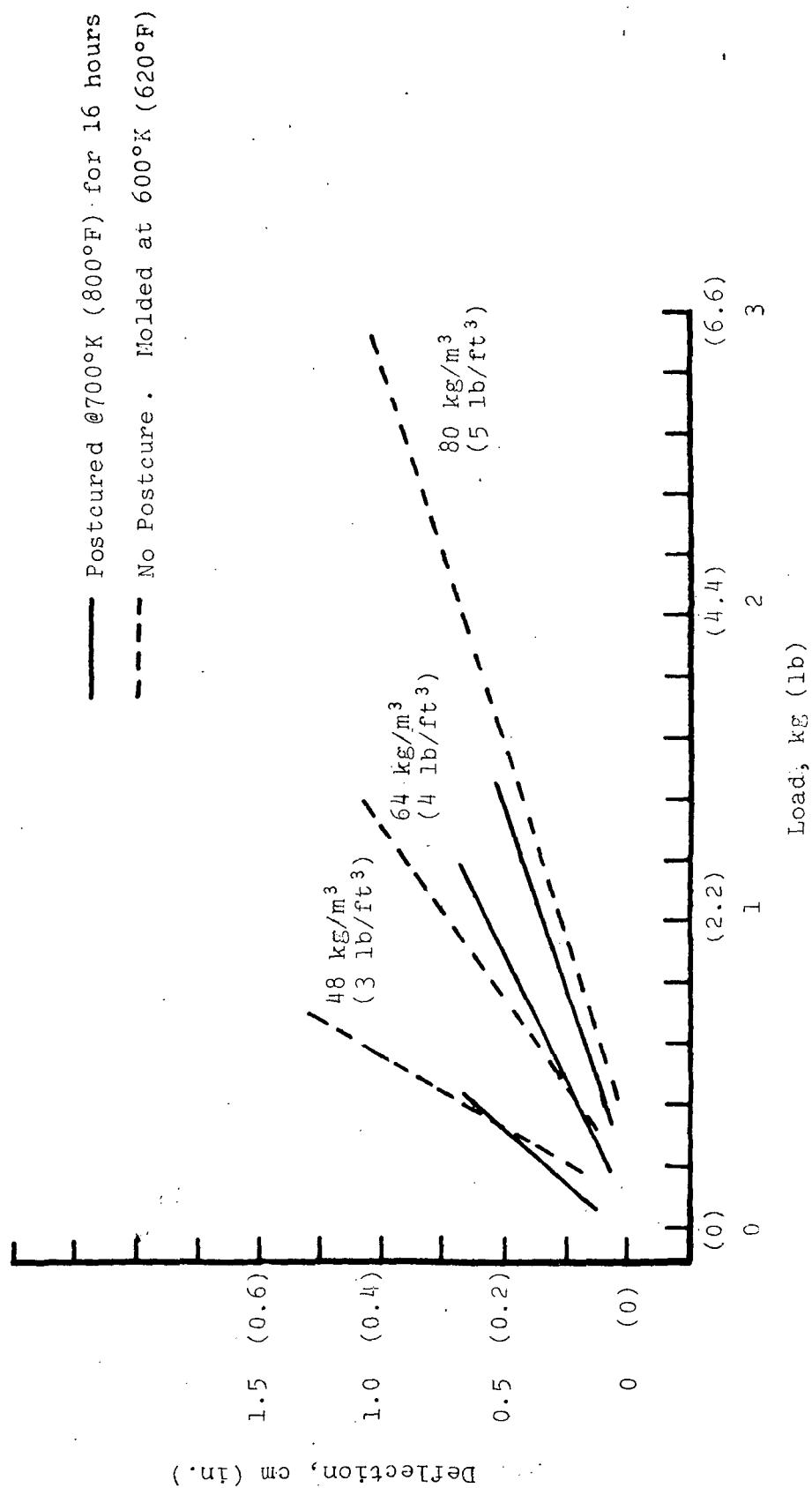


Figure 15. Load-Deflection Flexural Characteristics of Postcured and Unpostcured Polyimide Foams of Various Low Densities.

Table 7

EFFECT OF POSTCURE ON DROP IMPACT RESISTANCE<sup>(a)</sup>  
OF POLYIMIDE FOAMS<sup>(b)</sup>

Material		Impact Description	Rebound, %
Density <sup>(c)</sup> , (kg/m <sup>3</sup> ), (lb/ft <sup>3</sup> )	Cure		
48 kg/m <sup>3</sup> (3 lb/ft <sup>3</sup> )	-	very slight depression	48
	postcure <sup>(d)</sup>	2.5 mm (0.1 in.) depression	24
64 kg/m <sup>3</sup> (4 lb/ft <sup>3</sup> )	-	no significant depression	49
	postcure	1.8 mm (0.07 in.) depression	24
80 kg/m <sup>3</sup> (5 lb/ft <sup>3</sup> )	-	no depression	53
	postcure	0.76 mm (0.03 in.) depression	30

(a) 2.22 cm (0.866 in.) diameter steel ball, 44.7 g (0.0984 lb),  
 89 cm (34 in.) drop height, 2.54 cm (1 in.) thick samples.

(b) Molded at 600°K (620°F).

(c) Prior to postcure.

(d) 700°K (800°F) for 16 hours.

slight but was confined strictly to the geometry of the ball. Sub-surface damage was limited to a small circular cut, also defined by the geometry of the ball. Indeed, foams of even the lower densities would survive most hail storms and be useful for the intended purpose thereafter.

Resistance to Severe Acoustic Exposure: It is expected that the shuttle will be exposed to some severe acoustic vibration during the launch. It is, of course, important that the thermal insulation of the shuttle be able to withstand this acoustic environment, both cohesively and adhesively to its substructure. Accordingly, acoustic exposure tests were conducted on polyimide foams of various densities, both as-molded and following an elevated temperature postcure.

The densities of the unpostcured polyimide foams examined were 32, 48, 64, 80, 96 and 128 kg/m<sup>3</sup> (2, 3, 4, 5, 6 and 8 lb/ft<sup>3</sup>). The postcured foam examined had a density of 64 kg/m<sup>3</sup> (4 lb/ft<sup>3</sup>). The postcured specimen had been exposed to the stepwise temperature conditioning involving 64 hours at 644°K (700°F), followed by 16 hours at 670°K (750°F) and 2 hours at 700°K (800°F) in air. The test specimens were 29 cm x 29 cm x 2.5 cm (11.5 in. x 11.5 in. x 1 in.) panels of the polyimide foam mounted to a 1.6 mm (0.0625 in.) thick aluminum plate 31 cm (12.2 in.) square. The foam was bonded to the aluminum plate using an epoxy adhesive over the entire contacting surface. A bead of silicone adhesive was applied around the edges of the polyimide foam at the bond line to prevent edge lift-up. Holes were drilled in the edges of the aluminum plate to provide a means for connecting it to the test fixture.

The actual acoustic testing was done at McDonnell Aircraft (MCAIR) in St. Louis. Detailed test data are given in Appendix C. The acoustic exposure involved a "shuttle" frequency spectrum suggested by MCAIR as being representative of ultimate use. The minimum resistance that the foam needed to exhibit (as specified by NASA-Langley) was a 30-second exposure to this acoustic spectrum at an intensity of 162 dB. However, since this exposure had no effect on any of the specimens, the specimens were subjected to additional exposure intervals at 162 dB. Those that passed 6 minutes of this exposure were then exposed to 30-second intervals at 166 dB, a level which greatly exceeds expected shuttle exposure.

The test was conducted by first attaching the aluminum-bonded foam panels to the test fixture shown in Figure 16. This was a 15 cm (6 in.) deep rigid aluminum box filled with flexible urethane foam to "float" the test panel.

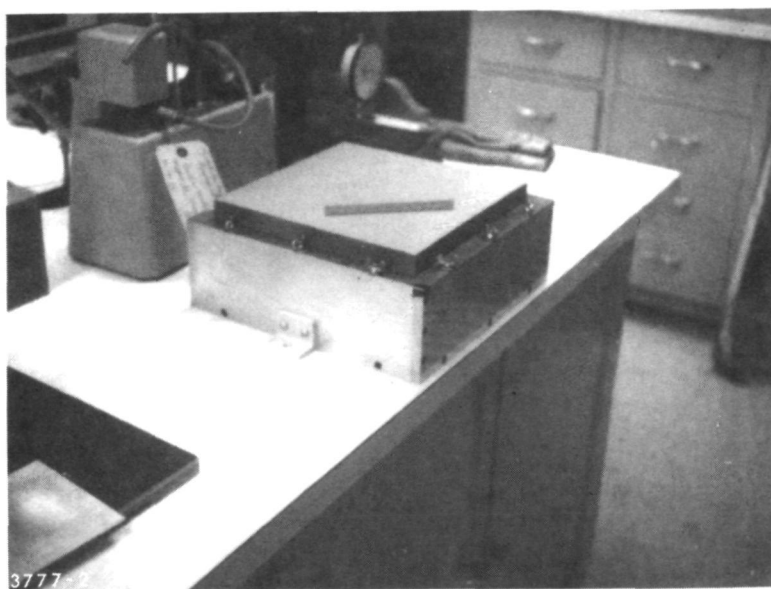


Figure 16. Polyimide Foam Panel Bonded to Aluminum Sheet and Mounted on Acoustic Load Fixture

Test results for the polyimide foams exposed to the simulated shuttle acoustic spectrum are given in Table 8, and illustrated in Figures 17 and 18.

It was shown that foams in all densities passed the minimum 30-second exposure at 162 dB. While small pieces of foam spalled from the surface of the 32 kg/m<sup>3</sup> (2 lb/ft<sup>3</sup>) foam in the second 30-second burst, all the higher densities resisted up to 6 minutes of the 162 dB exposure. The foams having densities of 48 and 64 kg/m<sup>3</sup> (3 and 4 lb/ft<sup>3</sup>) were then exposed to 30-second bursts at 166 dB. The first 30-second exposure at 166 dB resulted in a high degree of spalling of the 48 kg/m<sup>3</sup> (3 lb/ft<sup>3</sup>) foam from the panel. The foam having a density of 64 kg/m<sup>3</sup> (4 lb/ft<sup>3</sup>) passed the first 30-second burst at 166 dB, but some minor cracking and spalling occurred during the second 30-second burst (see Figure 18). In all cases, failure was attributed to the first bending mode vibration of the entire aluminum substrate-foam assembly.

The 64 kg/m<sup>3</sup> (4 lb/ft<sup>3</sup>) density post-cured foam did not pass the first 30-second exposure at 162 dB. However, this was attributed to an adhesive failure which was shown to be due to wicking of the adhesive into the foam which eliminated any degree of bonding to the aluminum. It is expected that the post-cured foam could easily pass the 162 dB acoustic exposure if properly bonded, but the test was not repeated.

The lowest density (32 kg/m<sup>3</sup>; 2 lb/ft<sup>3</sup>) polyimide foam passed the minimum acoustic exposure test, but it was eliminated from further consideration in the program because it was felt that it may represent a marginal material. Foams having densities of 48 kg/m<sup>3</sup> (3 lb/ft<sup>3</sup>) and higher will more than resist any anticipated shuttle acoustic exposure.

Surface Pressure and Shear (Erosion) Resistance: The thermal insulating material will be exposed to surface pressure and shear upon entry, though to a very low degree. Accordingly, the effect of high velocity air flow on the surface of both postcured and unpostcured polyimide foams was examined using a high pressure air line,  $5.5 \times 10^5$  N/m<sup>2</sup> (80 psi), and the nozzle sketched in Figure 19.

Maximum shear was generated by directing the air flow parallel to the foam surface. Maximum surface pressure was simulated using a 90° angle of the nozzle to the foam surface. Maximum combination of surface and shear pressure was generated using a 45° angle between the air flow and the surface. Exposures were for 15 seconds each. Additionally, the air stream was directed at the edge of the foam surface to amplify the severity of the exposure representative of inadvertently high exposed foam edge.

Table 8  
EFFECT OF SIMULATED SHUTTLE ACOUSTIC EXPOSURE ON POLYIMIDE FOAMS (a)

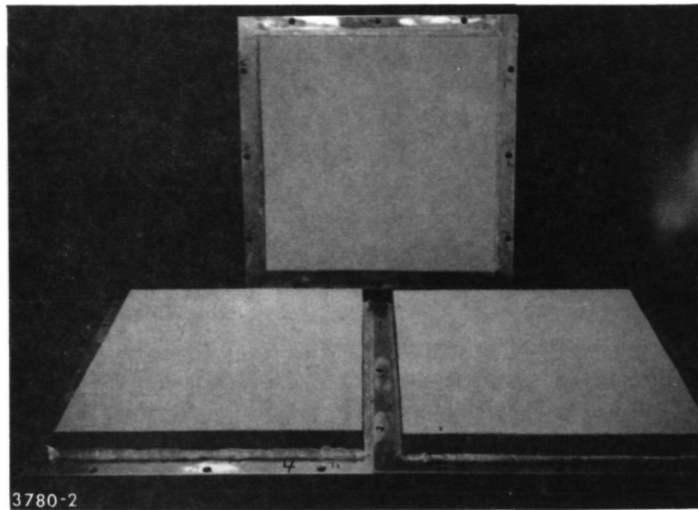
OF VARIOUS DENSITIES									
Failure at Indicated Intensity (dB) for Accumulated Exposure Time in Seconds									
Foam Density kg/m <sup>3</sup> (lb/ft <sup>3</sup> )	Foam Postcure	Accumulative Time, S Time Increments, S	162 dB						166 dB
			30	60	90	180	360	390	
32 (2)	none		30	30	30	90	180	30	420
48 (3)	none		N <sup>(b)</sup>	2 small pieces removed near edge	1 small piece removed	small cracks developed	terminated	(c)	-
64 (4)	none		N	N	N	N	N	almost complete failure	-
64 (4)	incremental up to 700°K (800°F)		N	N	N	N	N	N	partial failure
80 (5)	none		adhesive bond failure	-	-	-	-	-	-
96 (6)	none		N	N	N	N	N	-	-
128 (8)	none		N	N	N	N	N	-	-

(a) Panels 29 cm x 29 cm x 2.5 cm (11.5 in. x 11.5 in. x 1 in.) molded at 600°K (620°F) and bonded to a 0.16 cm (0.0625 in.) aluminum sheet. The aluminum sheet was attached to the acoustic load cell and placed within an acoustic horn operating at 166 dB utilizing a space shuttle frequency-intensity spectrum simulation.

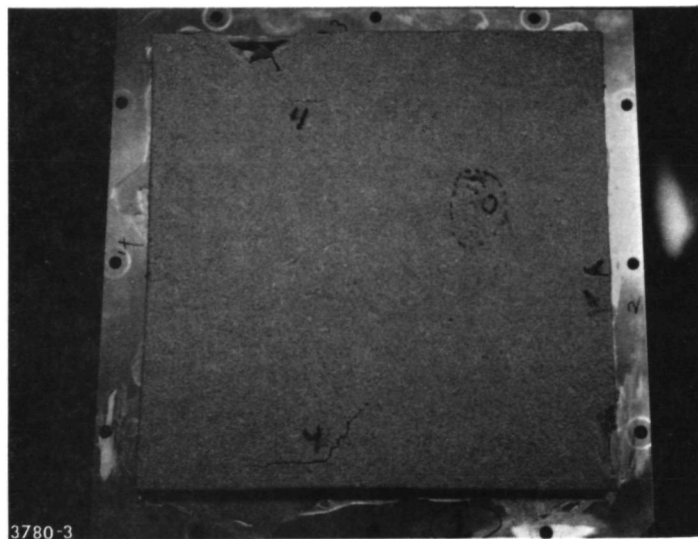
(b) N - no effect was observed.

(c) Test not performed for this increment.

(d) 64 hours at 644°K (700°F), 18 hours at 670°K (750°F) and 2 hours at 700°K (800°F)



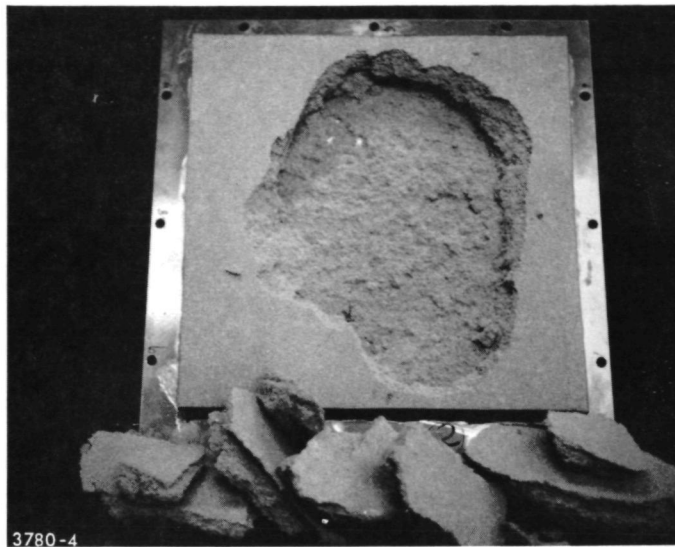
(a) 80, 96 and 128 kg/m<sup>3</sup> (5, 6, 8 lb/ft<sup>3</sup>) density foam after 6 minute exposure  
No observable effect shown.



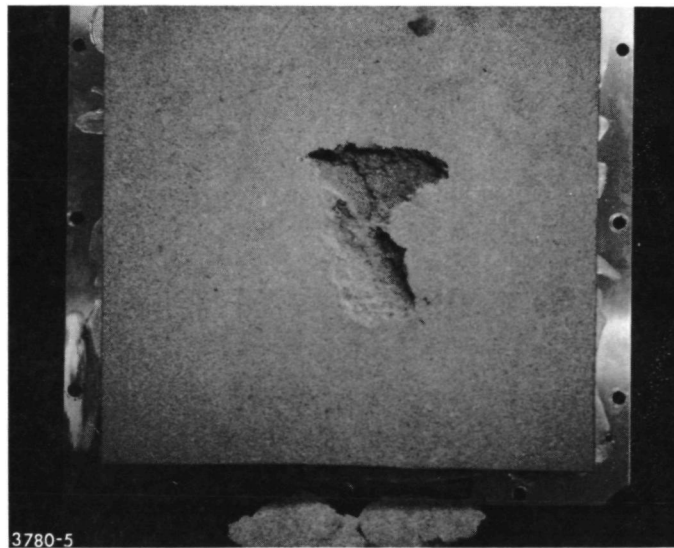
(b) 32 kg/m<sup>3</sup> (2 lb/ft<sup>3</sup>) density foam after 3 minute exposure. Note minor spalling.

Figure 17. Condition of Unpostcured Polyimide Foams Following Exposure to 162 dB Simulated Shuttle Acoustic Test





(a) 48 kg/m<sup>3</sup> (3 lb/ft<sup>3</sup>) density foam after 30 second exposure. Note large spalled area.



(b) 64 kg/m<sup>3</sup> (4 lb/ft<sup>3</sup>) density foam after 60 second exposure. No failure in first 30 second exposure.

Figure 18. Condition of Unpostcured Polyimide Foams Following Exposure to 166 dB Simulated Shuttle Acoustic Test in Addition to 6 minutes at 162 dB.

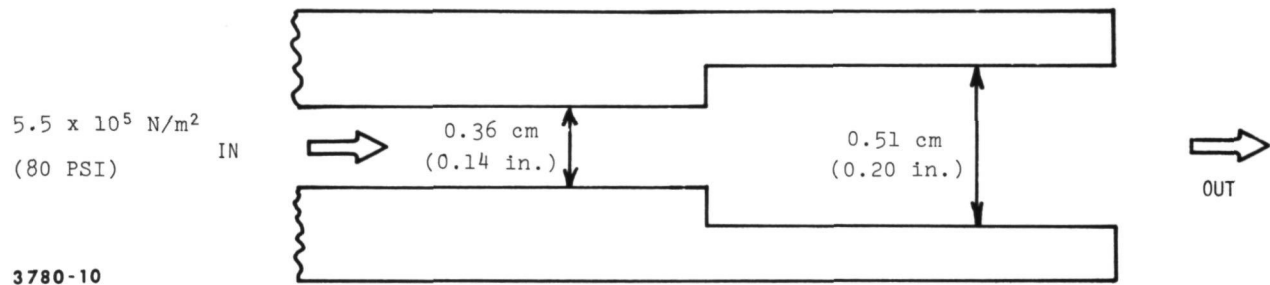


Figure 19. Schematic Drawing of Nozzle Used for High Velocity Air Shear and Surface Pressure Testing of Polyimide Foams

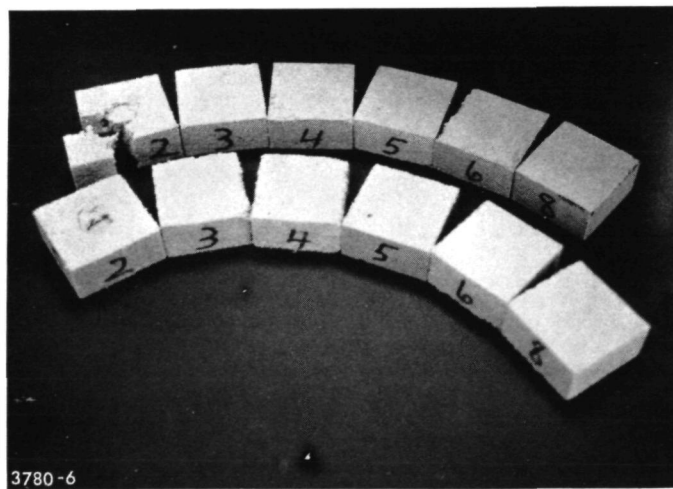


Figure 20. Results of Low Temperature (liquid nitrogen) Ball Drop Impact Tests on Polyimide Foams

The effect of the surface pressure and shear on the polyimide foam at 296°K (73°F) is illustrated in Table 9. Foams having densities from 48 to 80 kg/m<sup>3</sup> (3 to 5 lb/ft<sup>3</sup>) were examined, both as-molded and following a post-cure of 700°K (800°F) for 16 hours. The excellent resistance of the foam to shear (0°) and combinations of shear and pressure (45°) is illustrated. The increased resistance of the foam to shear and especially pressure due to postcuring is also illustrated. Resistance to maximum pressure conditions and edge erosion is very good for foam densities at or above 48 kg/m<sup>3</sup> (3 lb/ft<sup>3</sup>).

Characteristics of the polyimide foam at low and elevated temperatures: As a means to further amplify the highly desirable characteristics of the polyimide foam, some of its mechanical characteristics were determined at liquid nitrogen temperatures and at temperatures as high as 725°K (850°F). These included the low temperature impact, zero load thermal deflection, and high temperature deformation.

Low Temperature Impact Resistance: The impact resistance was determined at sub-ambient temperatures both in a manner similar to that described earlier for room temperature impact resistance and by empirical, non-standard drop tests.

The first impact test involved dropping a 2.2 cm (0.875 in.) diameter steel ball a distance of 90 cm (35 in.) onto foams that had been soaked in liquid nitrogen. Foams having densities from 32 up to 128 kg/m<sup>3</sup> (2 to 8 lb/ft<sup>3</sup>) were examined. The results (duplicate) are illustrated in Figure 20. Only the lowest density foam 32 kg/m<sup>3</sup> (2 lb/ft<sup>3</sup>) failed upon impact. Post-cured foams were not examined; they would be expected to have slightly poorer low temperature impact resistance.

The second impact test involved soaking 2.5 cm (1 in.) cubes in liquid nitrogen and then dropping them from a height of 1.3 meters (51 in.) onto a hard surface. When soaked, the foam weights were all independent of density. Both as-molded and post-cured foam samples were examined as a function of density.

The 32 kg/m<sup>3</sup> (2 lb/ft<sup>3</sup>) density foam completely crumbled upon impact. The 48 kg/m<sup>3</sup> (3 lb/ft<sup>3</sup>) foam broke into a number of larger pieces, and the 64 kg/m<sup>3</sup> (4 lb/ft<sup>3</sup>) foam broke into a few large pieces. The foams having densities ranging from 80 to 128 kg/m<sup>3</sup> (5 to 8 lb/ft<sup>3</sup>) all survived this drop impact test. Interestingly, while the postcured foams behaved in a similar manner, they appeared to be somewhat stronger. The postcure was 16 hours at 700°K (800°F). The postcured 48 and 64 kg/m<sup>3</sup> (3 and 4 lb/ft<sup>3</sup>) foam was slightly stronger. The postcured 80 kg/m<sup>3</sup> (5 lb/ft<sup>3</sup>) foam exhibited no observable failure.

Table 9

## EFFECT OF SURFACE PRESSURE AND SHEAR ON THE POLYIMIDE FOAMS AT 296°K (73°F)

Material		0°, Max. Shear		90°, Max. Pres.		45°, Shear & Pres.		Edge	
Density	(a)	Postcure	(b)	(Erosion)	(Erosion)	(Erosion)	(Erosion)	Erosion	Erosion
48 kg/m <sup>3</sup> (3 lb/ft <sup>3</sup> )		none		none	deep	slight		removed all of edge	
48 kg/m <sup>3</sup> (3 lb/ft <sup>3</sup> )		yes		none	moderate	slight		removed most of edge	
64 kg/m <sup>3</sup> (4 lb/ft <sup>3</sup> )		none		none	slight	very slight		slight	
64 kg/m <sup>3</sup> (4 lb/ft <sup>3</sup> )		yes		none	very slight	very slight		slight	
80 kg/m <sup>3</sup> (5 lb/ft <sup>3</sup> )		none		none	very slight	almost none		very slight	
80 kg/m <sup>3</sup> (5 lb/ft <sup>3</sup> )		yes		none	very slight	very slight		very slight	

(a) labeled density is that of unpostcured foam

(b) all molded at 600°K (620°F); those postcured exposed to 700°K (800°F) for 16 hours.

Zero Load Thermal Deflection Resistance: To illustrate the rigidity and strength of both postcured and uncured polyimide foams at elevated temperatures, unloaded flexural specimens (20 cm x 5 cm x 2.54 cm; 8 in. x 2 in. x 1 in.) were supported on 15 cm (6 in.) centers in a preheated oven. The deflection of this beam was recorded after 15 minutes exposure at the given temperature. Foams of three densities were examined.

The results of the zero load thermal deflection tests as a function of temperature are given in Table 10. All foams (all densities) exhibited little deflection at 590°K (600°F), just below the foam molding temperature. As expected, significant deflection was observed for the uncured specimens at temperatures above 600°K (620°F). Significantly, the postcured foams all exhibited little deflection at temperatures as high as 725°K (850°F), even though the postcure was to only 700°K (800°F). All specimens, however, did eventually fail in flexure at 750°K (900°F).

High Temperature Deformation (Creep) Resistance: The deformation resistance of both the uncured and postcured foams, ranging in densities from 48 to 80 kg/m<sup>3</sup> (3 to 5 lb/ft<sup>3</sup>), was examined by loading with relatively low weights at discrete elevated temperatures up to 725°K (850°F). The load was a 2.2 cm diameter (0.87 in.) steel ball weighing 45 grams (0.099 lb). The specimen was a 5 cm x 5 cm x 2.5 cm (2 in. x 2 in. x 1 in.) thick block of preheated foam. The results of this loading at various temperatures are given in Table 11. The uncured foams were substantially deformed even below the molding temperature of 600°K (620°F), while the postcured materials were undeformed even above the postcuring temperature of 700°K (800°F).

#### Structural Properties and Moisture Resistance of the Polyimide Foam

The various final requirements for the thermal insulation of the shuttle suggested that some additional characterization was needed. The open cell content, the moisture pick-up and the water vapor permeability of the polyimide foam were of interest. The open cell content of the foam is important because it affects the ability of the foam to survive radical changes in pressure (e.g. in going from atmospheric to the low vacuum of space) without exploding. Since the foam does have some open cell content, it is then important to determine how readily the foam picks up water, permeates it, and retains it.

Open cell content of the polyimide foam: The open cell content of the polyimide foams were determined using comparative density measurements with an air pycnometer. The open cell

# ZERO LOAD THERMAL FLEXURE TEST RESULTS FOR POLYIMIDE FOAMS

Material		Deflection (a) Cm (In.) at Indicated Temp. (b)°K (°F)					
Density (c)	Postcure (d)	589°K (600°F)	649°K (700°F)	700°K (800°F)	728°K (850°F)	755°K (900°F)	
48 kg/m <sup>3</sup> (3 lb/ft <sup>3</sup> )	none	0.30 (0.12)	2.6 (1.04)	-	-	-	Fail
	yes	0.30 (0.01)	0.08 (0.03)	0.08 (0.01)	0.05 (0.02)	-	Fail
64 kg/m <sup>3</sup> (4 lb/ft <sup>3</sup> )	none	0.1 (0.04)	0.48 (0.19)	0.76 (0.30)	1.2 (0.47)	-	Fail
	yes	0 (0.00)	0.03 (0.01)	0.03 (0.01)	0.03 (0.01)	-	Fail
80 kg/m <sup>3</sup> (5 lb/ft <sup>3</sup> )	none	0 (0.00)	0.20 (0.08)	0.33 (0.13)	0.51 (0.20)	-	Fail
	yes	0 (0.00)	0.05 (0.02)	0.05 (0.02)	0 (0.00)	-	Fail

- (a) 20.3 cm x 5.08 cm x 2.54 cm (8 in. x 2 in. x 1 in.) block, 15.2 cm (6 in.) support.
- (b) 15 minute heat and 15 minute cool cycle.
- (c) Density of foam molded at 600°K (620°F) with no postcure.
- (d) Postcure 700°K (800°F) for 16 hours.

Table 11

## HIGH TEMPERATURE DEFORMATION TEST RESULTS FOR POLYIMIDE FOAMS

Material Density (c)	Postcure (d)	Penetration (a), mm (in.), Temp. (b), °F		
		589°K(600°F)	644°K(700°F)	700°K(800°F) 728°K(850°K)
48 kg/m <sup>3</sup> (3 lb/ft <sup>3</sup> )	none	8.9 (0.35)	11 (0.43)	13-15 (0.51-0.59)
	yes	none	none	none
64 kg/m <sup>3</sup> (4 lb/ft <sup>3</sup> )	none	3.8 (0.15)	13 (0.51)	~18 (0.71)
	yes	none	none	none
80 kg/m <sup>3</sup> (5 lb/ft <sup>3</sup> )	none	-	14 (0.55)	18 (0.71)
	yes	-	none	none

(a) 2.22 cm (0.874 in.) diameter, 44.7 g (0.0984 lb) steel ball.

(b) 15 minute heating cycle.

(c) Density of foam molded at 600°K (620°F) with no postcure.

(d) Postcure 700°K (800°F) for 16 hours.

content was measured as a function of foam density and on foams both as-molded and following postcure. Results of the testing are shown in Figure 21.

The open cell content was, as expected, very high for these relatively low density foams and was inversely proportional to density. Significantly, the postcured foams exhibited an open cell content only 1% higher than that of the unpostcured foam. This was to be expected since weight is lost in postcuring.

Water pickup due to complete immersion: Water pickup of 48, 64 and 80 kg/m<sup>3</sup> (3, 4 and 5 lb/ft<sup>3</sup>) density foams was examined using 2.5 cm (1 in.) cubes immersed to a head of 2.5 cm (1 in.) for various periods of time. Both molded and postcured foams were examined. The results of this immersion test are given in Table 12.

The results show that the postcured polyimide foams become saturated with water within 1 minute, while the unpostcured foams were not saturated even after 20 hours. If water adsorption must be prevented, the postcured foams would probably require a covering or skin (although it is expected that altering postcuring conditions to eliminate the presence of oxygen may alter the surface characteristics and increase the hydrophobic character).

Removal of imbibed water from the polyimide foam: Since the postcured polyimide foams were found to imbibe water readily, it was important that the potential effect of this water on thermal insulation integrity, and on the ability to remove the water from the polyimide foam, be defined.

First, to illustrate the integrity of the foam to rapid removal of the entrained water, strips (2.5 cm x 2.5 cm x 10 cm; 1 in. x 1 in. x 4 in.) of both molded and postcured foams of a variety of densities were saturated with water and then shock loaded by being placed in a vacuum oven at 493°K (428°F). In all cases the water was removed quickly with no evidence of degradation to the cell structure or the bulk foam. Foams ranging in density from 48 to 80 kg/m<sup>3</sup> (3 to 5 lb/ft<sup>3</sup>) were examined. Additionally, when similar strips of water-saturated postcured foams were placed in a preheated 700°K (800°F) oven, no degradation was noted.

Larger blocks of foam (5 cm x 5 cm x 2.5 cm, 2 in. x 2 in. x 1 in.) were used to determine the rate at which water-saturated foams would dry under ambient conditions (294°K, 70°F and 50% RH). Foams of three densities were examined. The results, illustrated in Figure 22 show that all the water readily evaporated from the postcured foams in a matter of several days. (Note, in Figure 22, that the final data points represent the initial densities of the foam thus indicating that no residual moisture was present).



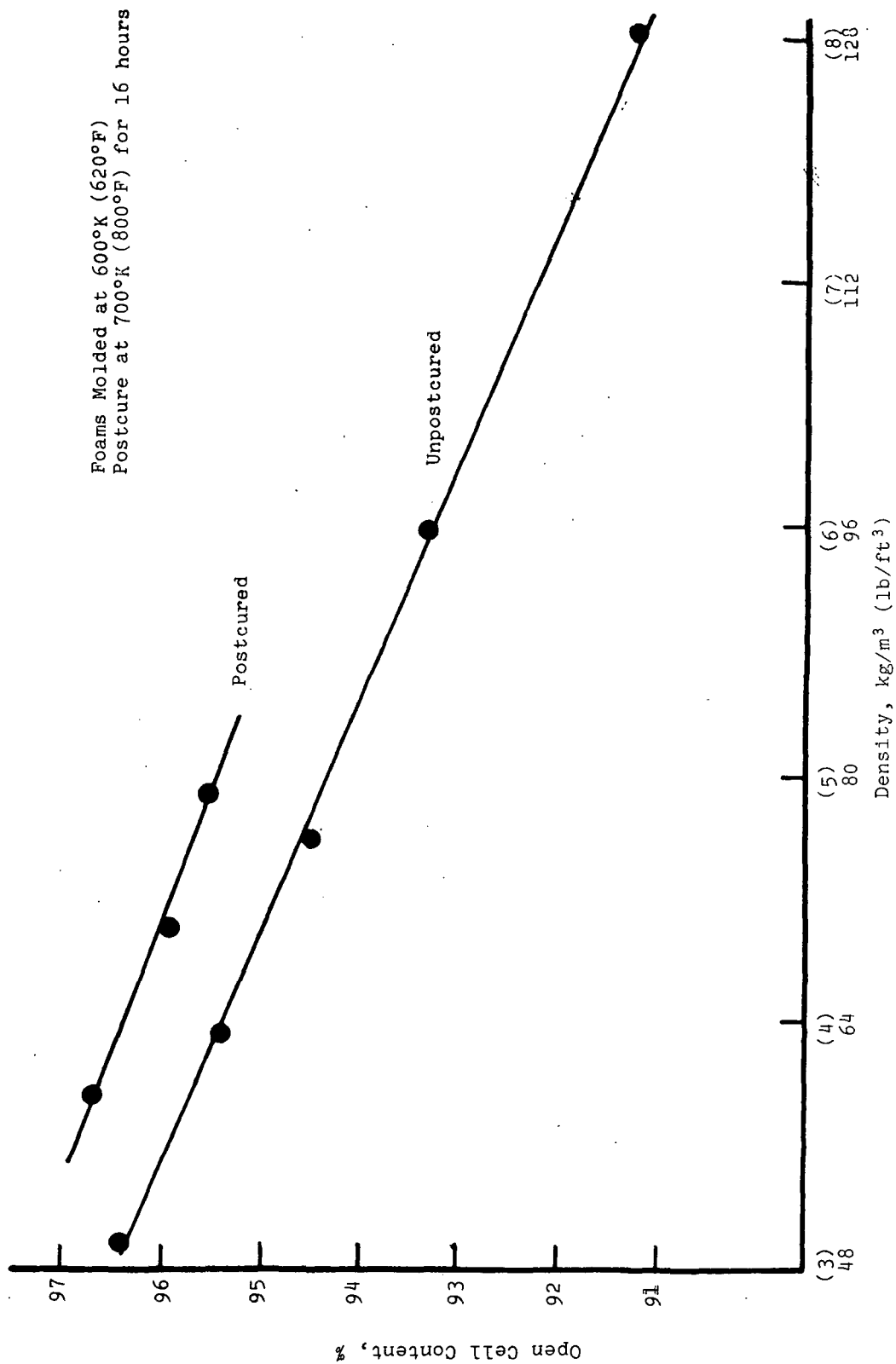


Figure 21. Open Cell Content as a Function of Density for Unpostcured and Postcured Polyimide Foams.

Table 12  
WATER ABSORPTION OF  
UNPOSTCURED AND POSTCURED POLYIMIDE FOAMS

Density Approx. kg/m <sup>3</sup> (lb/ft <sup>3</sup> )		Sample Weight, Wet, Grams <sup>(a)</sup>				
		Water Immersion Time <sup>(b)</sup>				
No Postcure <sup>(c)</sup>		0 Min	1 Min	10 Min	150 Min	1200 Min
48	(3)	0.8	1.1	1.7	1.7	1.5
64	(4)	0.9	1.5	1.5	1.7	1.8
80	(5)	1.5	2.1	2.1	2.3	2.4
<u>Postcured<sup>(d)</sup></u>						
48	(3)	0.7	12.4	12.9	13.1	13.2
64	(4)	0.8	13.4	13.4	13.5	13.5
80	(5)	1.1	13.4	13.5	13.5	13.7

(a) 2.5 cm (1 in.) cubes; lbs., divide by 454 g/lb.

(b) 294°K (70°F), 1.2 cm (0.5 in.) ave. head.

(c) Molded at 600°K (620°F).

(d) Postcured at 700°K (800°F) for 16 hours.

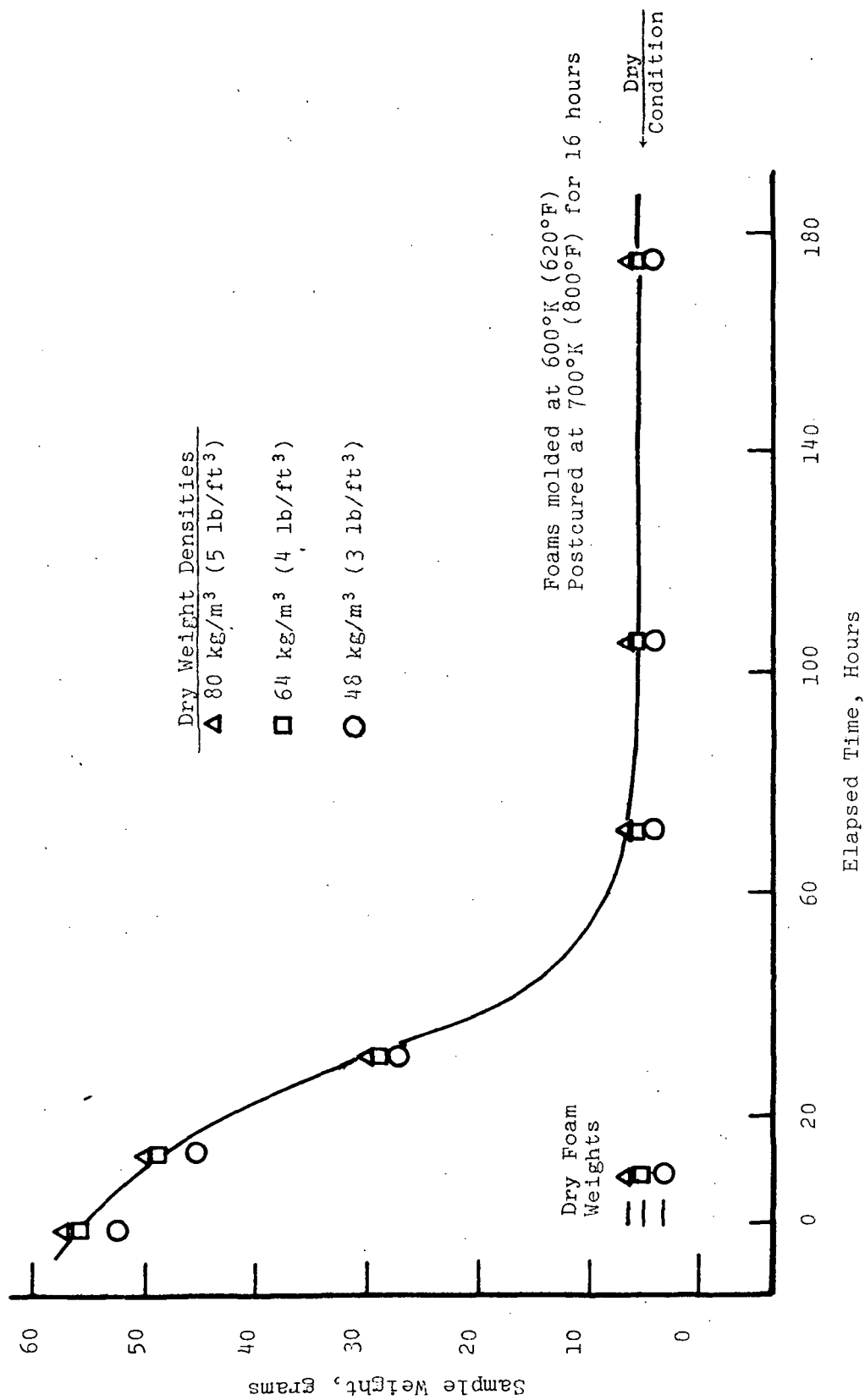


Figure 22. Water Removal from Saturated Postcured Polyimide Foams at 297°K (70°F).

Water vapor absorption: It is anticipated that the thermal insulation of the shuttle will be exposed to high humidities and a fair degree of radiation heating, and cooling while sitting on the ground prior to launch. Accordingly, the water vapor absorption of the foams is probably more important than the direct water pickup due to immersion.

Both as-molded and postcured foams were studied for water vapor adsorption by placing 2.5 cm x 2.5 cm x 10 cm strips (1 in. x 1 in. x 4 in.) in the 100% relative humidity chamber for six days. All sides were exposed; thus the surface-to-volume ratio was quite high. Foams with densities of 48, 64 and 80 kg/m<sup>3</sup> (3, 4 and 5 lb/ft<sup>3</sup>) were included. The maximum weight gain for the foams was 6% for as-molded foams and, unexpectedly, only 2% for postcured foams.

Accordingly, because of the comparatively small quantity of water absorbed, even at 100% RH, water vapor absorption is not believed to be a problem. Of course, skinning of the foams to reduce direct water pickup should also reduce this slight water vapor absorption.

Water vapor permeability: The water vapor transmission properties of 48 and 64 kg/m<sup>3</sup> (3 and 4 lb/ft<sup>3</sup>) postcured foams was determined using a technique based on ASTM D-96-66. Samples were approximately 3.8 cm (1.5 in.) in diameter and 1.3 cm (0.51 in.) thick. Testing conditions included an ambient of 294°K (70°F) and 58% RH. Weight loss versus time curves are shown in Figure 23, and the water vapor transmission, permeance, and average permeability of the postcured foams is given in Table 13.

As expected, the data indicate that the higher density foam exhibits a slightly lower water vapor transmission than the lower density material.

#### Cost Analysis of the Polyimide Foam in the Form of Thermal Protective Panels

A reasonable cost estimate for the postcured polyimide foam panels described in this report can only be projected using the present cost of materials and known conversion techniques. Because the starting material (Monsanto Company's RI-7271-01 polyimide foamable powder) is made on a custom basis in only very low volumes, it is very costly. Additionally, the preparation of panels has been conducted only in the laboratory. Thus, scale-up of the compression molding techniques forces a relatively high conversion cost.

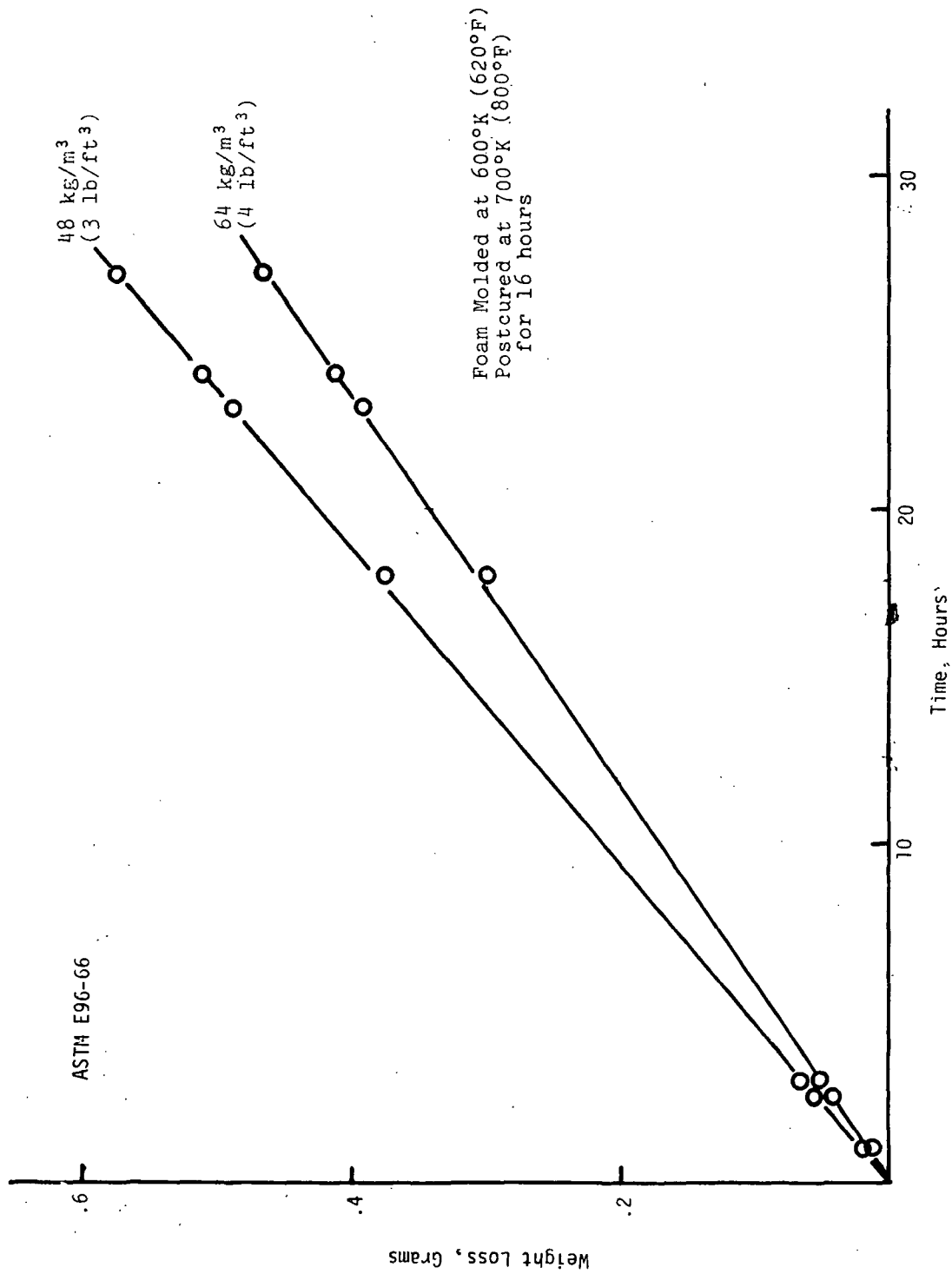


Figure 23. Water Vapor Transmission Through Postcured Polyimide Foams as a Function of Time (Based on ASTM E-96-66).

Table 13  
WATER VAPOR PERMEABILITY OF POSTCURED<sup>(a)</sup> POLYIMIDE FOAMS

<u>Density<sup>(b)</sup></u>	<u>Water Vapor<sup>(b)</sup> Transmission g/m<sup>2</sup>-24 hr</u>	<u>Permeance/ mm Hg, Metric Perms</u>	<u>Ave. Permeability Metric Perm-cm.</u>
48 kg/m <sup>3</sup> (3 lb/ft <sup>3</sup> )	446	0.475	0.576
64 kg/m <sup>3</sup> (4 lb/ft <sup>3</sup> )	359	0.392	0.482

(a) Postcured at 700°K (800°F) for 16 hours.

(b) Density of unpostcured foam molded at 600°K (620°F).

(c) 1.2 cm (0.47 in.) thick, 3.8 cm (1.5 in.) diameter.

The projected costs for the preparation of polyimide foam panels in various thicknesses and panel quantities is shown in Table 14. Typically,  $0.0929\text{m}^2$  ( $1\text{ ft}^2$ ) of  $1.27\text{ cm}$  ( $0.5\text{ in.}$ ) thick foam would cost \$38.35.

It is expected that both materials and manufacturing costs could be reduced eventually. Reduction in materials cost would require a much higher volume usage than shown here (the most material required for any of the items shown is 9320 lb, which is very low for a commercial product). There is no doubt that the materials cost could eventually drop nearly an order of magnitude, to \$22/kg (\$10/lb), but this would require the generation of business well above any projections here. It is expected that the manufacturing costs could be reduced significantly (at least half) by means of a developmental program. Thus, the potential for a \$269/ $\text{m}^2$  (\$25.00/ $\text{ft}^2$ ) material exists. The design, development, testing, and engineering program that would be required to maximize the performance of the polyimide foam and significantly reduce the present cost is projected to be in the neighborhood of \$500,000. This is a rough estimate which would require some additional cost analysis.

Table 14.

PROJECTED (a) COST INFORMATION FOR 64 kg/m<sup>3</sup> (4 lb/ft<sup>3</sup>) DENSITY

## POLYIMIDE (b) FOAM PANELS

Number (c) of Panels	Thickness, in.	Materials (d), lb	Total (e) Cost, \$	Cost/ft <sup>2</sup> (f), \$
100	0.25	11.7	11,200	112
100	0.50	23.3	12,200	122
100	1.0	46.6	13,700	137
100	2.0	93.2	16,300	163
1000	0.25	117	49,700	49.70
1000	0.5	233	57,000	57.00
1000	1.0	466	69,400	69.40
1000	2.0	932	92,700	92.70
10,000	0.25	1170	342,800	34.28
10,000	0.5	2330	383,500	38.35
10,000	1.0	4660	482,000	48.20
10,000	2.0	9320	665,800	66.58

(a) Based on 1974 material and labor costs and established manufacturing methods revised December 1974.

(b) Postcured to 700°F (800°F).

(c) 12 in. x 12 in. x indicated thickness (actual mfg. panels 24 in. x 24 in.)

(d) Amount of starting material required. Yield is 70%.

(e) Based on material cost of \$25/lb up to 1000 pounds and 18 \$/lb thereafter.

(f) 100 sq ft quantity does not include mold costs of ca. \$3000.



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## CONCLUSIONS

- (1) Polyimide foams exhibit excellent stability and thermal insulating characteristics up to 700°K (800°F).
- (2) Postcuring of the polyimide foams molded at 600°K (625°F) is required to establish stability (minimize weight and dimensional losses) to 700°K (800°F).
- (3) Polyimide foams of densities of 64 kg/m<sup>3</sup> (4 lb/ft<sup>3</sup>) or greater are highly resistant to anticipated shuttle exposures and environments up to 700°K (800°F) for at least 25 hours and for short times (less than 10 hours) to 725°K (850°F).
- (4) These polyimide foams will readily survive expected shuttle requirements of:
  - . Exposure to 162 dB acoustic loads for 30 seconds,
  - . Thermocycling from 144°K (-200°F) to 394°K (250°F),
  - . Surface pressures of  $5 \times 10^4$  N/m<sup>2</sup> (1000 lb/ft<sup>2</sup>; 0.5 atmosphere),
  - . Surface shears of 47.9 N/m<sup>2</sup> (1.0 lb/ft<sup>2</sup>),
  - . Surface heating to 533°K (500°F) for extended periods of time,
  - . Surface heating to 700°K (800°F) for a minimum of 25 hours, and
  - . Insulating ability on aluminum up to 640°K (700°F).
- (5) These polyimide foams will additionally:
  - . Provide a thermal conductivity of less than 8.3 watt/m·°k (1.2 Btu-in./hr-ft<sup>2</sup>-°F).
  - . Provide a thermal gradient of at least 220K° (400F°) across 2.3 cm (0.91 in.) with a surface temperature of 600°K (625°F).
  - . Resist acoustic loads of 166 dB for up to 60 seconds after 300 seconds exposure to 162 dB.

- . Resist drop impact at liquid nitrogen temperatures.
  - . Resist compressive, flexural, and impact loading expected during use and storage of the space shuttle.
  - . Exhibit no deflective creep up to 725°K (850°F).
  - . Exhibit little compressive creep up to 700°K (800°F).
- (6) While the unpostcured polyimide foam tended to pick up very little moisture in either high humidities or upon direct immersion in water, the postcured polyimide picked up 1600% of its weight upon complete immersion in water. Fortunately, however, the postcured polyimide foam picked up little moisture in high humidities (100% RH), and any water picked up was readily extracted upon standing at ambient conditions.
- (7) The cost of the polyimide foam in quantities of 929 m<sup>2</sup> (10,000 ft<sup>2</sup>) is projected at about \$38.00 per 0.0929 m<sup>2</sup> (1 ft<sup>2</sup>) for 1.27 cm (1/2 in.) thickness. This is because of the developmental nature of both the material and process. It is expected that this projected cost could be reduced drastically (halved) following a process development program, or when made in significantly higher volumes.
- (8) Polyimide foams having densities around 48 kg/m<sup>3</sup> (3 lb/cu ft) are expected to have marginal mechanical characteristics for the shuttle application. However, it is expected that further process development could be used to qualify material of this density for the shuttle application. Foams in the 32 kg/m<sup>3</sup> (2 lb/cu ft) density range lack adequate tensile strength.

## APPENDIX A

### PREPARATION OF THE POLYIMIDE FOAM MOLDINGS

MRC scientists developed a moldable, resilient polyimide foam for NASA, MSC under Contract NAS-9-12246. This foam is tougher and more usable than prior polyimide systems, which were either non-homogeneous or friable or both. These foams are prepared from a commercially available polyimide foamable powder (Monsanto RI-7271-01) primarily through physical modification to produce a bulk molding compound.

The molding compound is prepared from the commercial low density polyimide powder (RI-7271-01) which is first foamed to ca.  $16 \text{ kg/m}^3$  ( $1 \text{ lb/ft}^3$ ) density at  $450^\circ\text{K}$  ( $340^\circ\text{F}$ ) and then cured at  $570^\circ\text{K}$  ( $570^\circ\text{F}$ ). This foam is then shredded (not ground) into approximately 1/4-inch chunks having a bulk density of about  $8 \text{ kg/m}^3$  ( $0.5 \text{ lb/ft}^3$ ). The resulting raw material is thermoplastic, deformable, and fusible at temperatures in the range of  $598^\circ\text{K}$  ( $610^\circ\text{F}$ ). The preparation of the foam molding compound is shown in Figure A-1. The molding technique involves the compaction of this bulky, shredded, B-staged foam product into a mold, followed by fusion at  $600^\circ\text{K}$  ( $610^\circ\text{F}$ ), cooling, and removal of the part. Filling a mold with shredded foam is shown in Figure A-2. Precompressing the shredded foam in the mold is shown in Figure A-3. The foam is then fused in a hot air circulating oven and removed as shown in Figure A-4. Slabs of molded resilient polyimide foam are produced in this way ranging in densities from 32 to  $128 \text{ kg/m}^3$  ( $2\text{-}8 \text{ lb/ft}^3$ ).

This process for molding the polyimide foam can be used to produce a resilient, fine-textured, open-celled product in any density in the range between 32 and  $160 \text{ kg/m}^3$  ( $2\text{-}10 \text{ lb/ft}^3$ ). The polyimide foam in this range of densities has a tough skin imparted during the molding process. If necessary, the integrity of the skin can be further improved by hot ironing at the appropriate temperature after the part is removed from the mold. Foam slabs or contoured parts can be produced readily with this process, provided that an appropriate mold (e.g. chrome-plated steel) which will take the heat and modest pressure encountered during the curing operation is used. Obviously, the density is directly controlled by the amount of foam charged to the mold and the compression ratio.



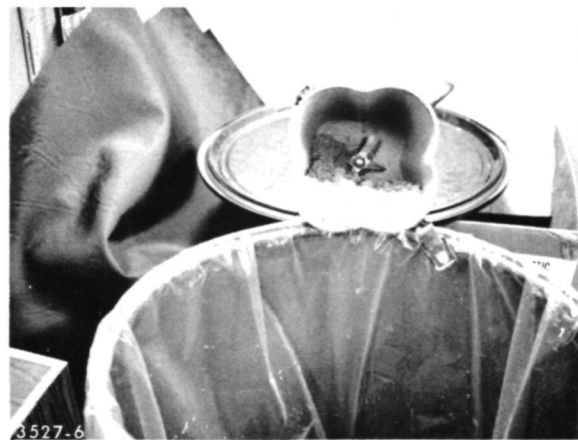
(a) Weighing RI-7271-01 Powder



(b) Foaming and Curing  
RI-7271-01 Powder

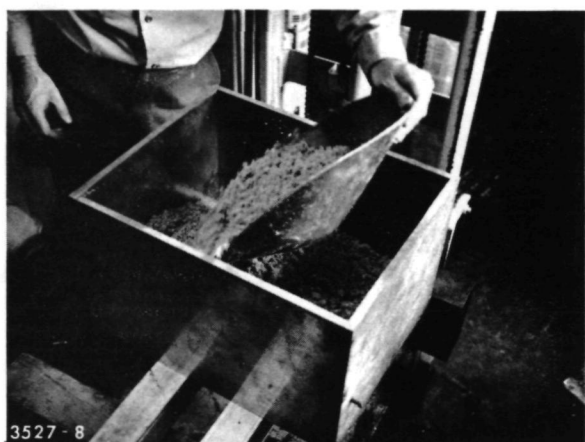


(c) Shredding, Cured Molding Compound



(d) Shredded, Foamed Cured  
RI-7271-01 Powder

#### A-1. Preparation of Polyimide Foam Molding Compound

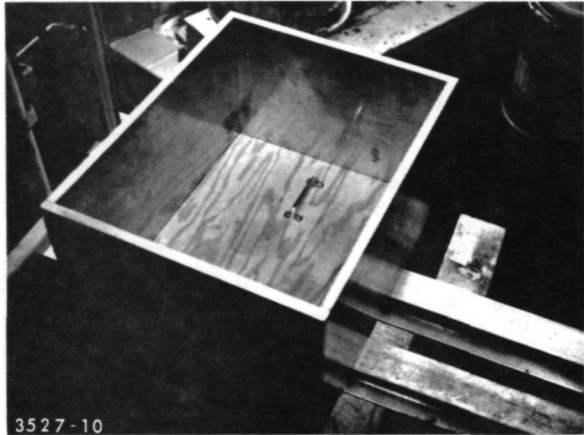


(a) Filling Mold with Shredded Foam

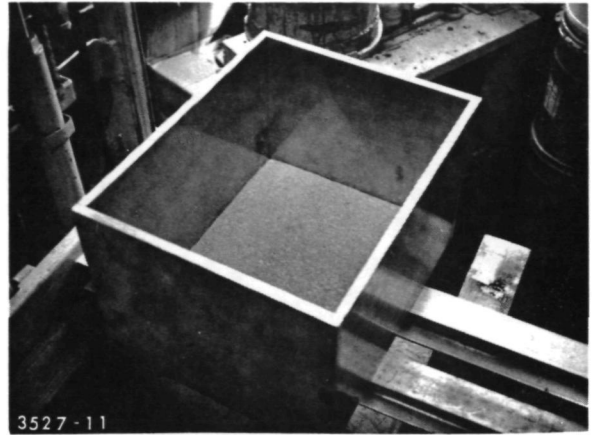


(b) Shredded Foam in Mold

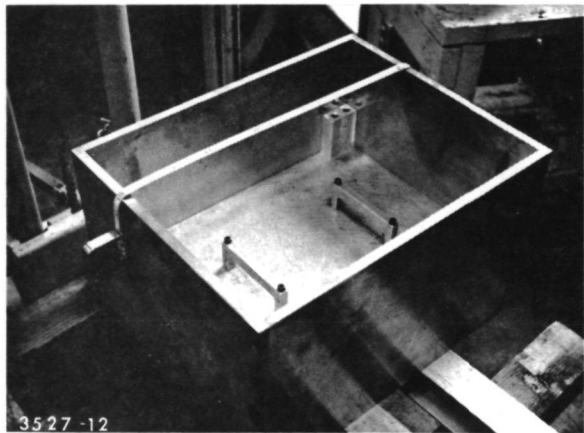
A-2. Filling of Mold with Polyimide Foam Molding Compound



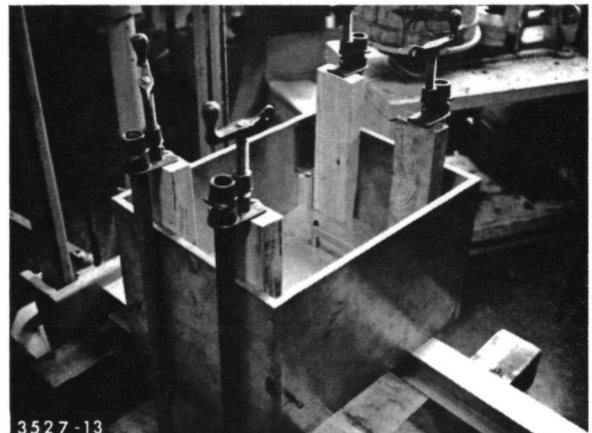
(a) Predensifying Shredded Foam



(b) Predensified Shredded Foam

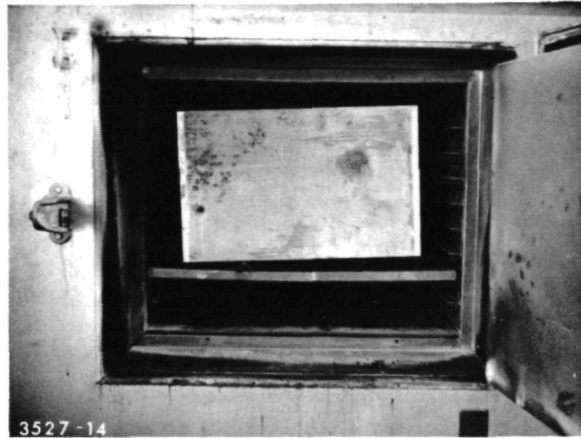


(c) Mold Top in Place



(d) Final Closing and Densifying of Shredded Foam

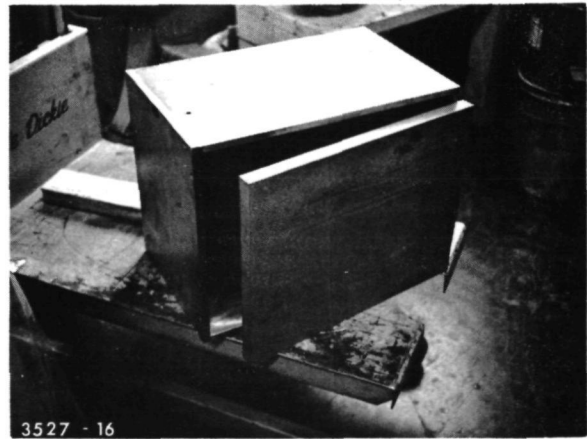
### A-3. Compacting of Polyimide Foam Molding Compound



(a) Fusion of Shredded Foam in Oven



(b) Mold Tilted for Removal of Part from Bottom



(c) Molded Resilient Polyimide Foam Part

#### A-4. Molding and Part Removal



Polyimide foams of this type will withstand prolonged heating at temperatures up to 570°K (570°F) without prohibitive loss in physical properties. Similar foams have a thermal k factor of 0.032 W/m-°K (0.22 Btu/hr/sq ft/°F/in.) at 296°K (73°C) in a density of 10 kg/m<sup>3</sup> (0.6 lb/ft<sup>3</sup>). Furthermore, these polyimide foams are very resistant to burning in air, or even in an oxygen-enriched atmosphere. If burned with a separate source of fuel, the polyimide foams produce little or no smoke or fumes and no known noxious combustion products (those normally encountered include CO<sub>2</sub>, water, N<sub>2</sub> and traces of other materials including carbon monoxide).

Also of some pertinence to the proposed application, polyimide foams of this type adhered to aluminum panels can withstand direct heating with a 1300°K (1800°F) fuel flame for one hour with only gradual loss of about 25% of the foam weight, while holding the temperature rise of the aluminum plate on the back side of the foam to less than 475°K (400°F).

APPENDIX B  
TEST REPORTS ON THE THERMAL CONDUCTIVITY  
OF POLYIMIDE FOAMS

*MCDONNELL AIRCRAFT COMPANY*

*Saint Louis, Missouri 62166*

18 July 1974

Monsanto Research Company  
1515 Nicholas Road  
P. O. Box 8, Station B  
Dayton, Ohio 45407

Attention: Mr. George L. Ball III - Research Group Leader

Subject: Thermal Conductivity Testing

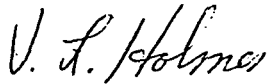
Enclosure: (1) TM 256.3358 dated 18 July 1974

Gentlemen:

Thermal conductivity of the two density Monsanto Research Company foam specimens was determined by the Materials and Physics Laboratories of the McDonnell Aircraft Company from 14 June to 8 July 1974.

The tests were performed utilizing a guarded hot-plate type calorimeter at points between ambient temperature and 400°F using the procedure outlined in ASTM C-177. A more detailed description of the test procedure is included in Encl (1). A list of the calibrated McDonnell equipment used during the test is available upon request.

Very truly yours,



V. L. Holmes  
Test Engineer  
McDonnell Aircraft Company



J. C. Bass  
Laboratory Project Engineer  
McDonnell Aircraft Company

VLH:JCB:jv

# Technical Memorandum

## ENGINEERING LABORATORIES

NO. TN 256.3358

DATE 18 July 1974

REV. \_\_\_\_\_

SUBJECT THERMAL CONDUCTIVITY OF MONSANTO RESEARCH

COMPANY FOAM SPECIMENS

T/WR NO. 700-789

MODEL NO. \_\_\_\_\_

TEST DEPT. 256

TEST STARTED 14 June 1974

TEST COMPLETED 8 July 1974

- ☒ PRIME DEPARTMENT FINAL REPORT  
☐ SUPPORT DEPARTMENT REPORT  
☐ LIMITED TR REPORT  
☐ INTERIM TEST REPORT NO.

PREPARED BY

*Vernon Holmes*  
V. L. Holmes, Test Engineer  
Applied Physics Laboratory

APPROVED BY

*L. E. McCrary*  
L. E. McCrary, Senior Group Engineer  
Physics Laboratories

### DISTRIBUTION:

NAME	DEPT.
A. H. Bay	256
J. C. Bass	250
G. L. Ball-Monsanto Research Company	
D. L. Kummer	E457
H. J. Siegel	247
Eng. Library	209

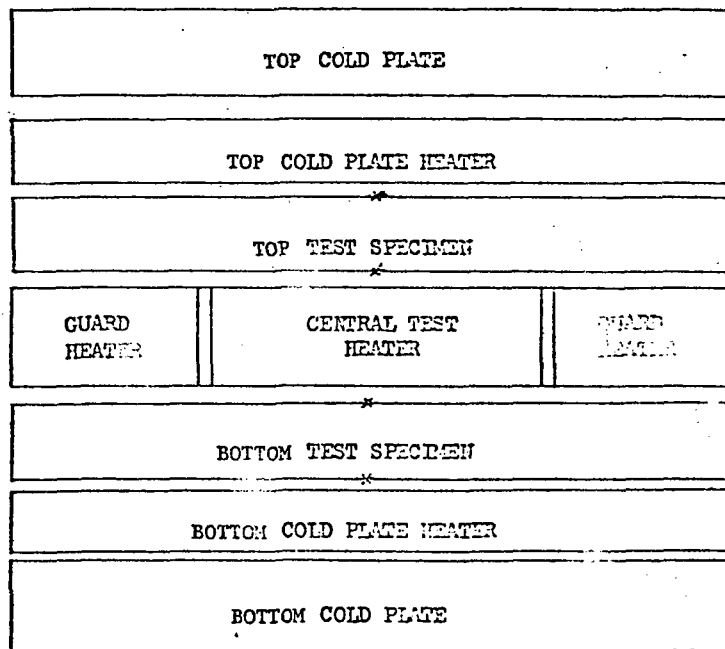
- The purpose of this test was to determine the thermal conductivity of two Monsanto Research Company foam specimens at mean temperatures between ambient and 650°F.
- Monsanto Research Company supplied two foam specimens<sup>(1)</sup> for evaluation having nominal densities of 3 and 6 pounds per cubic foot (PCF). Each specimen was eight inches in diameter and one-inch thick.
- The thermal conductivity of the foam specimens were measured in air at atmospheric pressure in a guarded hot plate type calorimeter following the general procedures outlined in ASTM C-177. The guarded hot plate calorimeter used a 4-inch diameter flat central heater guarded by a flat 4.2-inch ID, 8-inch OD guard heater. The central/guard heater was sandwiched between two identical test specimens which were sandwiched between cold plate heaters with water-cooled cold plates on the outside surfaces as shown schematically in Figure 1. The heaters attached to the inside surface of the cold plates permitted control of the specimens' mean temperature and temperature differential across the sample.

A differential thermocouple averaged the temperature difference at several points around the central-guard heater interface. The differential thermocouple was attached to a temperature controller to maintain the guard within 2°F of the temperature of the central heater. Radial heat flow from the central test section was reduced to a negligible value with this system.

- (1) Test discussions with Mr. Ball suggest the foam is a polyimide material.

**MCDONNELL AIRCRAFT COMPANY**

3. (Continued)  
Thermocouples were installed on each side of the test specimen. Since the thermocouple could not be physically attached to the foam specimens, the thermocouples were laid in grooves in the hot and cold plate heaters as stated in ASTM C-177.
4. To establish each desired test mean temperature, the power was adjusted to the central heater several times and then left for several hours to reach a steady state temperature distribution through the composite test stack. After temperature equilibrium had been established, temperatures were measured using a K-3 potentiometer. An ammeter and voltmeter were used to measure the current supplied to the central heater and the voltage drop across the heater itself. The power to the central heater was calculated from these data.
5. Since compression of the insulation was to be minimized for this test the top cold plate was supported by springs which allowed only a small load to be imposed on the insulation. For the test on the 3 PCF foam the heater was also supported using springs. The thickness of the insulation was measured before and after testing. During testing the composite specimen-heater stack height was monitored to record any compression versus temperature that might occur.
6. Tables 1 and 2 gives the temperature difference across the specimen, specimen mean temperatures, total power supplied to the central heater, average thickness of the two specimens, and the calculated thermal conductivity of the foam specimen. For both foam densities the bottom specimen was compressed more than the top specimen due to the weight of the central heater but very little difference was noted in the temperature difference across the top and bottom specimen. Figure 1 shows the thermal conductivity of the 3 and 6 PCF foams as a function of mean temperatures. Since the specimens did compress at high temperatures, the accuracy of the thermal conductivities are within  $\pm 8\%$  for temperatures below  $450^{\circ}\text{F}$  and  $\pm 12\%$  for the range  $450$  to  $650^{\circ}\text{F}$ .



\* THERMOCOUPLES

FIGURE 1 - SCHEMATIC OF THE SPECIMEN  
INSTALLATION IN THE GUARDED HOT PLATE CALORIMETER

FIGURE 1

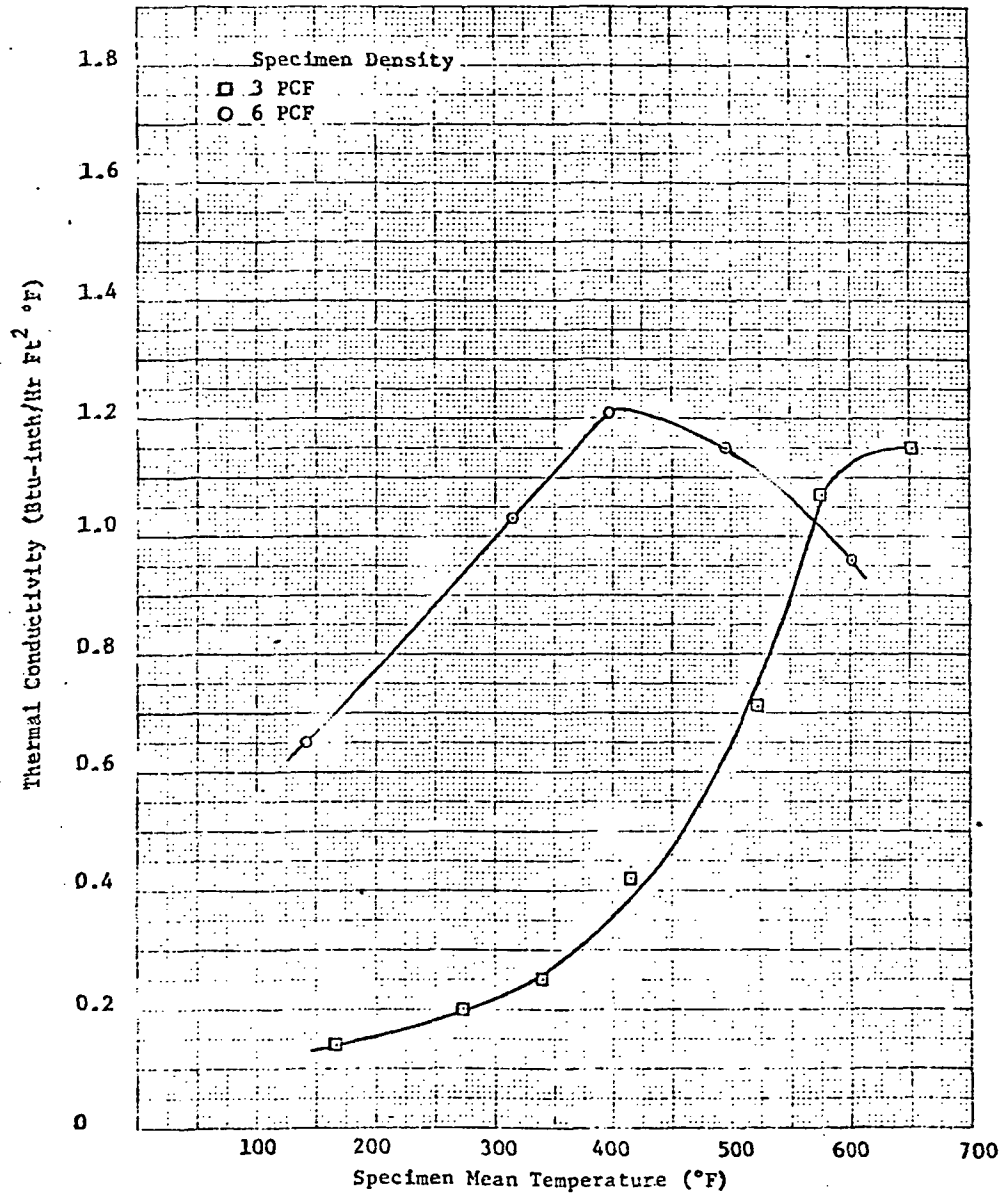


FIGURE 2 - THERMAL CONDUCTIVITY OF FOAM SPECIMENS VERSUS MEAN TEMPERATURE

FIGURE 2

TABLE 1 - TEMPERATURE, POWER, THICKNESS, AND THERMAL  
CONDUCTIVITY FOR THE 514 PCF FOAM SPECIMEN

Data Point No.	ΔT Across Specimen		Specimen Mean Temperature			Power to Central Heater (Watts)	Average Specimen Thickness (Inch)	Thermal Conductivity $\frac{\text{Btu-Inch}}{\text{Hr-Ft}^2\text{-}^\circ\text{F}}$
	Bottom (°F)	Top (°F)	Bottom (°F)	Top (°F)	Avg. (°F)			
1	104	99	102	141	144	3.39	1.00	0.65
2	108	97	103	326	332	5.39	1.00	1.03
3	89	97	92	396	394	5.67	1.00	1.21
4	93	100	96	492	491	5.80	0.97	1.15
5	120	126	123	600	602	7.09	0.85	0.96

TABLE 1

TABLE 2 - TEMPERATURES, POWER, THICKNESS, AND THERMAL CONDUCTIVITY FOR THE THREE PCF FOAM SPECIMEN

Data Point No.	$\Delta T$ Across Specimen		Specimen Mean Temperature		Power to Central Heater (Watts)	Average Specimen Thickness (Inch)	Thermal Conductivity $\frac{\text{Btu-inch}}{\text{hr-ft}^2\text{-}^\circ\text{F}}$
	Bottom ( $^\circ\text{F}$ )	Top ( $^\circ\text{F}$ )	Avg. ( $^\circ\text{F}$ )	Avg. ( $^\circ\text{F}$ )			
1	134	140	137	166	0.98	1.00	0.14
2	110	120	115	272	1.16	1.00	0.20
3	106	100	103	339	1.33	1.00	0.25
5	108	109	109	523	4.56	0.87	0.71
6	77	80	79	576	5.37	0.80	1.07
7	112	88	100	652	7.65	0.77	1.15

TABLE 2



# Technical Memorandum

## ENGINEERING LABORATORIES

NO. TM. 256.3508

DATE 10 December 1974

REV. \_\_\_\_\_

SUBJECT THERMAL CONDUCTIVITY OF MONSANTO RESEARCH

COMPANY FOAM SPECIMENS

T/WR NO. 700-329 MODEL NO. Outside Sales

TEST DEPT. 256 TEST STARTED 5 November 1974

TEST COMPLETED 20 November 1974

- ☒ PRIME DEPARTMENT FINAL REPORT  
☐ SUPPORT DEPARTMENT REPORT  
☐ LIMITED TR REPORT  
☐ INTERIM TEST REPORT NO.

PREPARED BY

Vernon L. Holmes  
V. L. Holmes, Test Engineer  
Applied Physics Laboratory

APPROVED BY

Leon E. McCrary  
L. E. McCrary, Senior Group Engineer  
Physics Laboratories

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J. C. Bass	250
G. L. Ball Monsanto	
Research Corporation	
D. L. Kummer	EA57
H. J. Siegel	247
Eng. Library	209

1. The purpose of this test was to determine the thermal conductivity of a Monsanto Research Corporation foam specimen at mean temperature between ambient and 750°F.
2. Monsanto Research Corporation supplied two eight-inch diameter discs one-inch thick for evaluation. The thermal conductivity of the foam specimen was measured in air at atmospheric pressure in a guarded hot plate type calorimeter following the general procedures outlined in ASTM C-177. The description of the test set up and test procedure was given in TM 256.3358 (Thermal Conductivity of Monsanto Research Company Foam Specimens) for the evaluation of two foam specimens earlier in 1974 (TR 700-789). The test set up and test procedure used in this test was identical to that used in TR 700-789 except that compression of the specimen was prevented by the use of quartz spacers.
3. Table 1 gives the temperature difference across the specimen, specimen mean temperatures, total power supplied to the control heater, average thickness of the two specimens, and the calculated thermal conductivity of the foam specimen. Figure 1 shows the thermal conductivity of the foam as a function of mean temperatures. The thermal conductivities are within  $\pm 8\%$ .

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4. To achieve a mean temperature higher than 549°F (676°F hot face temperature), a slight power adjustment to the central and cold plate heaters (see TM 256.3358) was made. These higher temperatures resulted in degradation of the foam and a slow continuous rise in specimen mean temperature overnight. The test was terminated because equilibrium could not be achieved. After slowly heating overnight for 17 hours, the hot face temperature had reached 897°F.

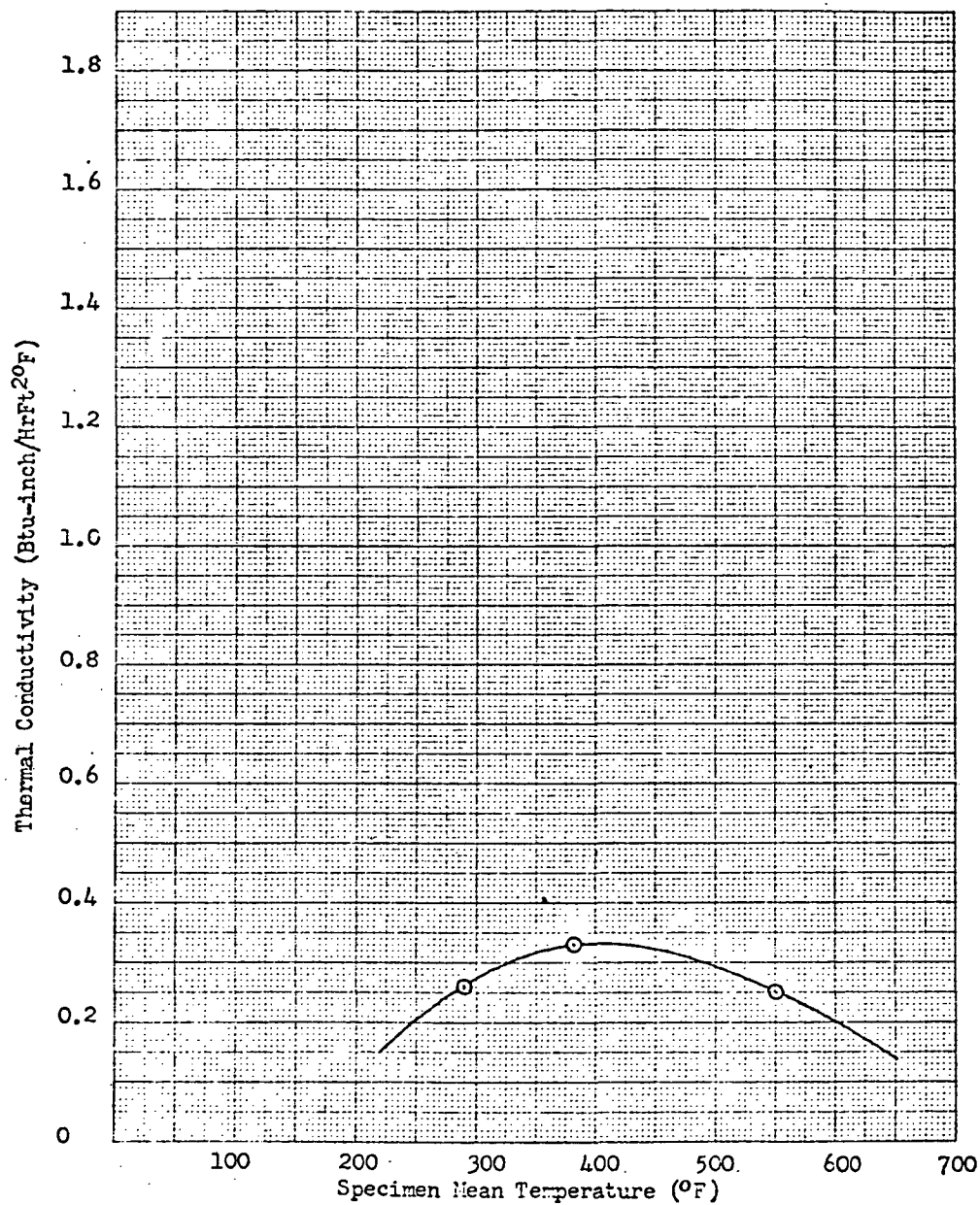


FIGURE 1 - THERMAL CONDUCTIVITY OF FOAM  
SPECIMEN VERSUS MEAN TEMPERATURE  
MCDONNELL DOUGLAS CORPORATION

FIGURE 1

TABLE 1 - TEMPERATURE, POWER, THICKNESS, AND  
THERMAL CONDUCTIVITY FOR THE FOAM SPECIMEN

Data Point No.	$\Delta T$ Across Specimen			Specimen Mean Temperature			Power to Central Heater (Watts)	Average Specimen Thickness (Inch)	Thermal Conductivity $\frac{\text{Btu-Inch}}{\text{Hr-Ft}^2 \text{ OF}}$
	Bottom (OF)	Top (OF)	Avg. (OF)	Bottom (OF)	Top (OF)	Avg. OF			
1	159	166	162	291	286	289	2.17	1.00	0.26
2	147	160	153	386	377	382	2.65	0.99	0.33
3	252	258	255	552	547	549	3.28	0.98	0.23

TM 256.3508  
TR 700-629

TABLE 1

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# APPENDIX C

## ACOUSTIC LOAD TEST RESULTS ON POLYIMIDE FOAMS

### Technical Memorandum

ENGINEERING LABORATORIES

NO. TR 253.1138

DATE 6 November 1974

REV. ---

SUBJECT ACOUSTIC TEST OF SIX PANEL SPECIMENS FOR

MONSANTO RESEARCH COMPANY

T/WR NO. 700-788 MODEL NO. Outside Sales

TEST DEPT. 253 TEST STARTED 12 June 1974

TEST COMPLETED 12 June 1974

- ☒ PRIME DEPARTMENT FINAL REPORT  
☐ SUPPORT DEPARTMENT REPORT  
☐ LIMITED TR REPORT  
☐ INTERIM TEST REPORT NO.

PREPARED BY A. A. Stewart  
A. A. Stewart, Senior Engineer

APPROVED BY R. W. Merkel  
R. W. Merkel, Sr. Group Engineer  
Structures & Dynamics Laboratories

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J. Loffingwell	<u>A</u>
R. W. Merkel	253
A. A. Stewart	253
E. C. Stuckman	250
Eng. Library	209
<u>A</u> Monsanto Research Company	

1. Acoustic testing was conducted for six panel specimens furnished by Monsanto Research Company. Testing was performed by the Structures and Dynamics Laboratory of the McDonnell Aircraft Company on 12 June 1974.
2. Each panel was subjected to a grazing incidence acoustic environment at an overall sound pressure level of 162 dB. The acoustic spectrum is presented in Figure 1. Two of the test panels were subjected to additional acoustic tests at an overall level of 166 dB. A test log indicating the panel designation, test level, test duration, and test results is presented in Table 1.

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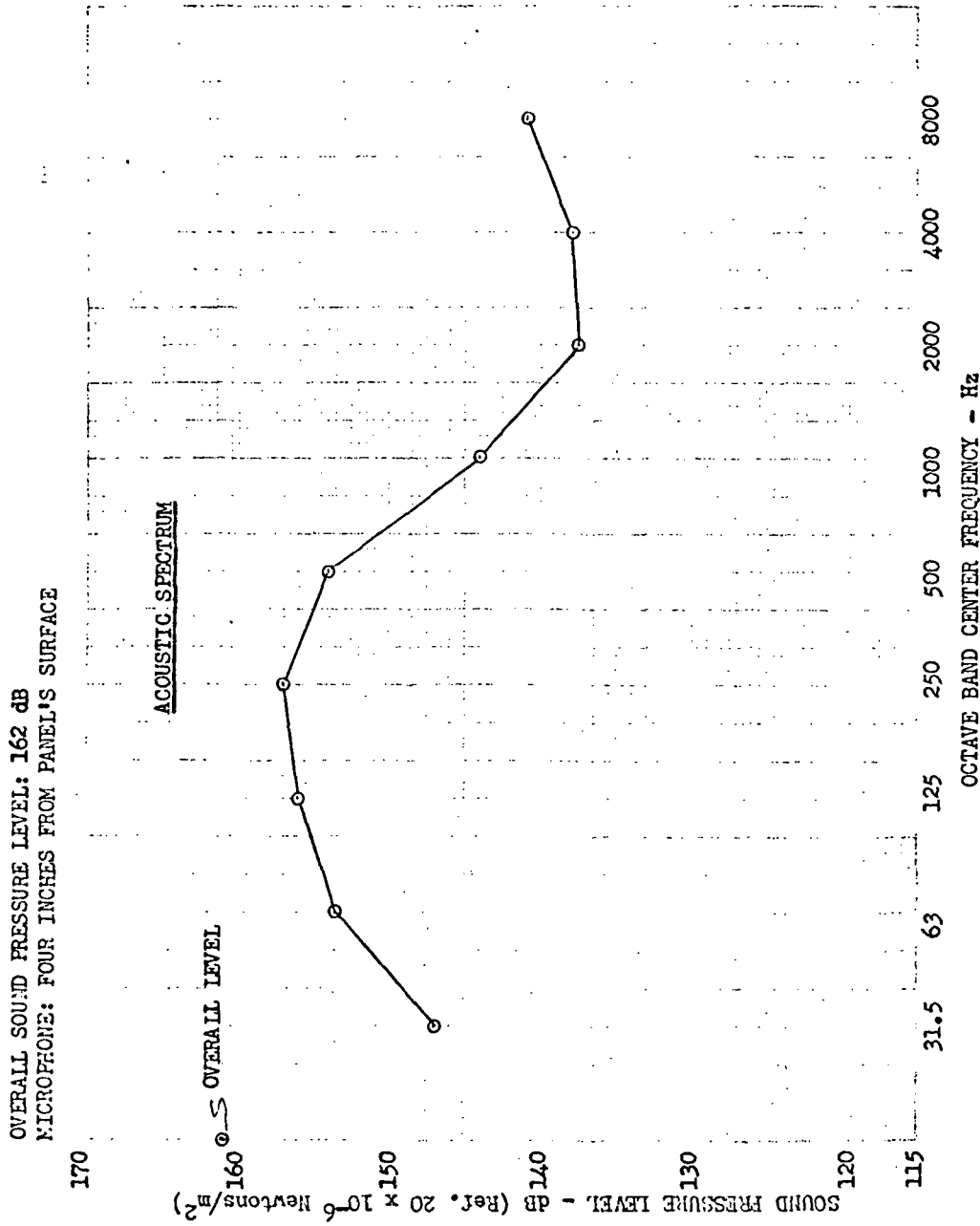


FIGURE 1 - OCTAVE BAND SPECTRUM ANALYSIS

MCDONNELL DOUGLAS CORPORATION

FIGURE 1

TABLE 1 - TEST LOG OF ACOUSTIC TEST  
PERFORMED ON MONSANTO PANELS

<u>PANEL DESIGNATION</u>	<u>TEST LEVEL (dB)</u>	<u>DURATION (Seconds)</u>	<u>RESULTS/REMARKS</u>
168822-1	162	180	No apparent deterioration after 30-seconds of testing. Inspection after 60 seconds of testing revealed minor panel deterioration. Deterioration continued for the remaining 90 seconds of this test.
168822-6	162	360	No failure and/or deterioration noted as a result of this test.
168822-5	162	360	No failure and/or deterioration noted as a result of this test.
168822-4	162	360	No failure and/or deterioration noted as a result of this test.
168822-3	162	360	No failure and/or deterioration noted as a result of this test.
	166	60	No failures noted after 30-seconds; however, complete failure was noted after 60-seconds of testing.
168822-2	162	360	No failure and/or deterioration noted as a result of this test.
	166	30	Complete failure occurred when panel was subjected to 166 dB level for 30-seconds.

TABLE 1



# Technical Memorandum

## ENGINEERING LABORATORIES

NO. 253.1142

DATE 18 November 1974

REV. 1

SUBJECT ACOUSTIC TEST OF MONSANTO RESEARCH CORPORATION

THERMAL PROTECTION MATERIAL

T/WR NO. 700-829 MODEL NO. Outside Sales

TEST DEPT. 253 TEST STARTED 1 November 1974

TEST COMPLETED 1 November 1974

- ☐ PRIME DEPARTMENT FINAL REPORT  
☒ SUPPORT DEPARTMENT REPORT  
☐ LIMITED TR REPORT  
☐ INTERIM TEST REPORT NO.

PREPARED BY L. F. Bares  
L. F. Bares, Test Engineer

APPROVED BY R. W. Merkel  
R. W. Merkel, Sr. Group Engineer  
Structures & Dynamics Laboratories

### DISTRIBUTION:

NAME	DEPT.
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L. F. Bares	253
J. C. Bass	250
R. W. Merkel	253
Eng. Library	209
<u>A</u> Monsanto Research Corporation	

1. The purpose of this test was to expose a thermal protection insulation specimen to a representative Space Shuttle acoustic environment. The test specimen was supplied by Monsanto Research Corporation.
2. The specimen was a 12 x 12 x 1 inch piece of thermal protection material bonded to a thin aluminum panel. The panel was attached to a rigid fixture with twelve wing nut fasteners. The inside of the fixture was filled with styrofoam.
3. The specimen/fixture assembly was placed on an isolation pad on the floor of a progressive wave chamber for the test. The acoustic input energy was monitored with a microphone mounted 12 inches above the specimen.
4. The specimen was subjected to an acoustic environment of 162 dB with an octave band distribution of Figure 1 for 30 seconds. Post-test inspection revealed that the thermal protection material had separated from the panel and was lying in pieces on the acoustic room floor. A photograph of the failed specimen is presented in Figure 2. It appears that the bond between the thermal protection material and the aluminum panel was inadequate.
5. This test was conducted by the Structures & Dynamics Laboratory of the McDonnell Aircraft Company on 1 November 1974.

**MCDONNELL AIRCRAFT COMPANY**

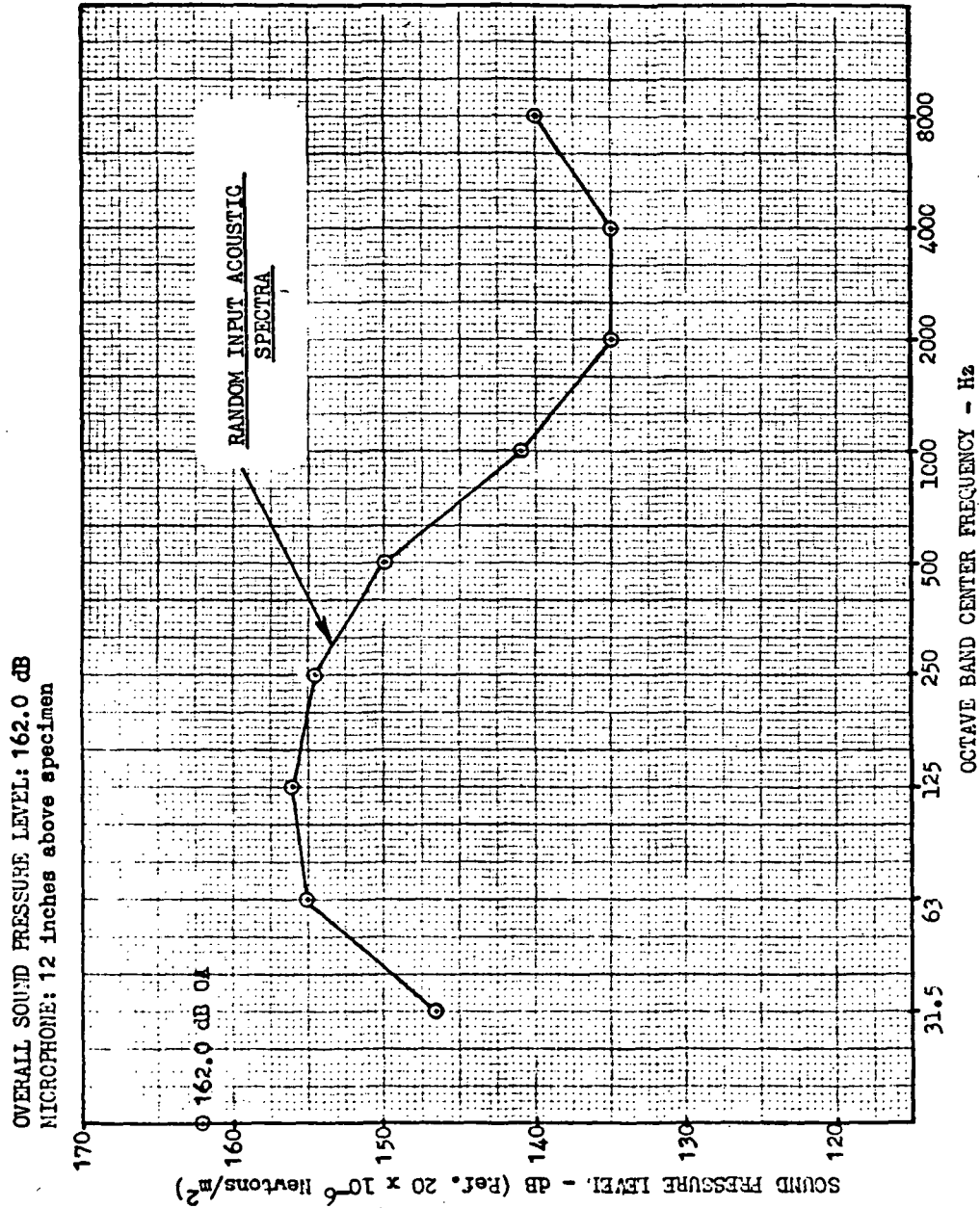


FIGURE 1 - OCTAVE BAND SPECTRUM ANALYSIS

MCDONNELL DOUGLAS CORPORATION

FIGURE 1

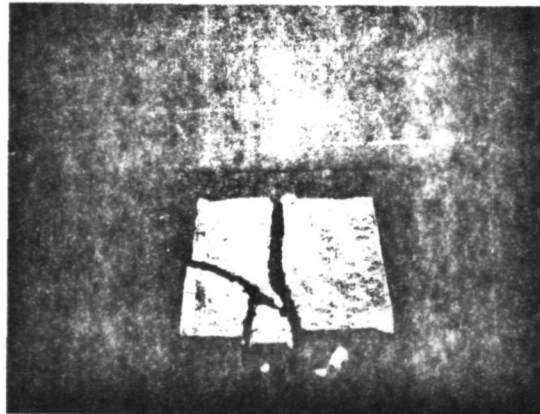
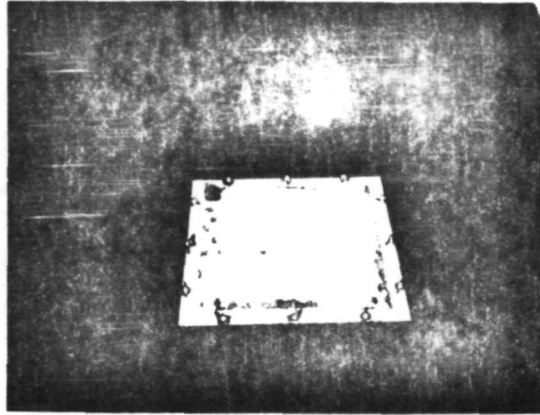


FIGURE 2 - MONSANTO RESEARCH CORPORATION THERMAL PROTECTION  
SPECIMEN AFTER 162 dB ACOUSTIC TEST

DPL-5 0641

MCDONNELL DOUGLAS CORPORATION

FIGURE 2

3

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Monsanto Data Sheet, Skybond<sup>®</sup> RI-7271 Foamable Polyimide Powders, Pub. No. 6253.

Jerrard and McNeill, "Theoretical and Experimental Physics," Chapman and Hall, London, 1960. (Unguarded Hot Plate Method for Thermal Conductivity Screening Test).

ASTM Designation: D1621-64, "Standard Method of Test for Compressive Strength of Rigid Cellular Plastics."

ASTM Designation: C 356-60 (67), "Standard Method of Test for Linear Shrinkage of Preformed High Temperature Thermal Insulation Subjected to Soaking Heat."

ASTM Designation: D 1622-63, "Standard Method of Test for Apparent Density of Rigid Cellular Plastics."

ASTM Designation: C 447-64, Standard Method for Determining the Maximum Use Temperature of Preformed High-Temperature Insulation."

ASTM Designation: D 1623-64, "Tensile Properties of Rigid Cellular Plastics."

ASTM Designation: D 282-69, "Water Absorption of Rigid Cellular Plastics."

ASTM Designation: C 355-64, "Water Vapor Transmission of Thick Materials."

ASTM Designation: C-177-63, "Standard Method of Test for Thermal Conductivity of Materials by Means of the Guarded Hot Plate."

ASTM Designation: C 203-58 (65), "Standard Method of Test for Breaking Load and Calculated Flexural Strength of Preformed Block-Type Thermal Insulation."